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GALENIC PHARMACY

GALENIC PHARMACY

A Practical Handbook to the Processes of the British Pharmacopœia

SPECIALLY DESIGNED AS A

GUIDE FOR THE STUDENT OR APPRENTICE

AND ADAPTED AS A

WORK OF REFERENCE FOR THE PHARMACIST,
CONTAINING CONCISE NOTES ON PILL-COATING,
COMPRESSED TABLETS, PASTILS, SURGICAL DRESSINGS,
AND MANY OTHER NON-OFFICIAL PREPARATIONS,
WITH A GLOSSARY OF TERMS

BY

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ILLUSTRATED BY SEVENTY-SIX ENGRAVINGS

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PREFACE

In a paper recently published in the 'Pharmaceutical Journal' Mr. Joseph Ince remarks as follows:—"Nothing is more difficult than to set the boundaries of pharmacy, of which chemistry is the mainspring, while other sciences lend tributary assistance. There still exists a distinct plot of ground called pharmacy, which it may task one's best energies to cultivate aright, and *he who imagines a knowledge of certain allied sciences to be alone sufficient will be mournfully disappointed.*"

While such works as Attfield's or Fownes' Chemistry, Bentley's or Prantl and Vines' Botany, and Deschanel's or Ganot's Physics have amply provided the student with instruction in these sciences, he has been left almost without help in their application to the PRACTICE OF PHARMACY.

Such being the case, no apology is needed for the appearance of this manual, which it is hoped will satisfy the want which has so long been felt to exist.

In order that the apprentice or student may obtain a thorough insight into the manipulation of drugs for the production of preparations therefrom, there is no substitute for laboratory work, none whatever; he is therefore urged to work out examples of every class of preparations, and on *no account to omit those which are marked with an asterisk.* By conscientiously following out this rule he will become so grounded in his art that he will be able successfully to meet the many difficulties which will be certain to present themselves in the course of his daily work as a pharmacist.

The calling of Pharmacy presents a field of work which is equalled by few others; it should be the object of every Pharmacist to so prepare each drug that its medicinal activity may be presented in its most potent, palatable, and digestible form, whilst rejecting unnecessary or injurious ingredients, thus to contribute towards the advancement of the noble art of medicine, and so assist in the alleviation of the sufferings of the human race—truly an object worthy his best endeavours!

The Author has great pleasure in expressing his thanks to those who have kindly lent him blocks for many of the illustrations, or in other ways assisted in the production of this volume, viz. the Editor of the 'Pharmaceutical Journal;' Messrs. Southall Bros. and Barclay, Birmingham; Messrs. Townson and Mercer, Bishopsgate Street, E.C.; Brinjes and Goodwin, Fieldgate Street, E.; S. Maw, Son, and Thompson, Aldersgate Street, E.C.; Burroughs and Wellcome, Holborn Viaduct, E.C.; Christy and Co., Lime Street, E.C.,; Fletcher and Russell, Warrington; E. R. and F. Turner, Engineers, Ipswich; and Werner and Pfleiderer, Queen Victoria Street, E.C.

He will also welcome any suggestions which may serve to make the work more thoroughly practical, which communications should be addressed to the publishers.

R. A. CRIPPS.

BIRMINGHAM;
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GALENIC PHARMACY

INTRODUCTION

Pharmacy.—The art of applying the laws of Chemistry and Physics to the preparation of drugs in a form suitable for administration in medicine—this is *Pharmacy.*

A *Pharmacopœia* is a book containing a selection of the most approved medicines, with directions for their preparation, or tests for their purity.

The British Pharmacopœia—which was published first in 1864—was made authoritative by Act of Parliament in 1862–3, and declared to supersede the Pharmacopœias of the London, Edinburgh, and Dublin Colleges of Medicine. Since that time the national Pharmacopœia has been re-written in 1867 (with additions in 1874), and again in 1885—additions to the last having been made in 1890.

Drugs and preparations which are included in the British Pharmacopœia are said to be *official* in this country; those not included are *non-official.*

Officinal preparations are preparations commonly kept in the pharmacist's shop, but not authorised by the national Pharmacopœia (from *officina,* a shop).

A *Dispensatory* is a commentary upon a Pharmacopœia, supplemented by notices of non-official remedies.

Drugs may be broadly divided into two groups: (1) substances of definite chemical composition, such as metallic salts and alkaloids; and (2) crude drugs of vegetable and animal origin, possessing somewhat indefinite and variable characters. The study and preparation of drugs of the first group constitute a large portion of the science and art of *Chemistry;* whilst the study of the growth and characters of the latter come within the domains of *Botany, Zoology,* and *Materia Medica.*

Divisions of Pharmacy.—The art of Pharmacy has been commonly divided into three sections, viz. *Chemical pharmacy, Galenical pharmacy,* and *Extemporaneous pharmacy* or *Dispensing.*

Chemical pharmacy is really little more than chemistry, and treats of the preparation of such inorganic salts and definite organic compounds as are used in medicine.

Galenical or galenic pharmacy is the art of producing preparations of natural substances for medical use, whilst still remaining mere mixtures of more or less active as well as inactive constituents. It is so called after Galen, a celebrated medical writer of the second century, who practised the art of healing by means of vegetable products.

Extemporaneous pharmacy, or the art of *Dispensing,* is closely allied to that of galenic pharmacy. It consists mainly of the mixing or compounding of the various galenic and chemical preparations according to the prescriptions of the physician.

In the opinion of the author it is advisable to restrict the use of the term *Pharmacy* to the second of these sections, the first being included in the study of chemistry, the third forming a separate branch of study.

Scope of this Work.—On this account this work is mainly a treatise on galenic pharmacy, the first section being only described by equations of the chemical processes employed, and a general description of the preparation of alkaloids and other definite compounds obtained from natural drugs, as these methods involve much that is strictly pharmaceutical; and the third section is represented only by notices of such preparations as are common to both galenical and extemporaneous pharmacy.

The object of the 'Handbook of Galenic Pharmacy' is to assist the student in gaining such a practical knowledge of the art of *Pharmacy* proper (galenic pharmacy) as shall enable him to produce satisfactory preparations of any given drug, and at the same time to gain a thorough grasp of the *raison d'être* of the various processes employed.

The author believes that this object will be best attained by means of a gradual progression from the most simple operations of pharmacy—such as the mixing of powders—up to such complex processes as the preparation of standardised extract of nux vomica; he has therefore adopted such a system, arranging the preparations of the British Pharmacopœia in sections, *according to the methods employed in their manufacture;* and has introduced such explanations of chemical and physical laws as are necessary, although this work is in no sense intended to supplant works on chemistry and other sciences, the study of which should be carried on simultaneously with pharmacy.

The student who is familiar with the Elements of Chemistry and Botany, and is able to answer the questions at the close of Chapters I and II, may proceed at once to Chapter III.

CHAPTER I

PRELIMINARY CHEMICAL NOTES

In order to render the study of Pharmacy intelligible to the student, it is necessary to devote a short space to the first principles of the science of Chemistry.

Elements and Compounds.—All known substances are composed of one or more simple bodies or *elements*, so called because in the present stage of chemical science it has been found impossible to decompose them into simpler bodies. *Compounds* are substances consisting of two or more elements.

Chemistry is the science which treats of the interchange of elements in compounds.

Symbols.—It is convenient to represent the elements by symbols, the symbol chosen being in most instances the first letter of the Latin name of the element, *e. g.* O = oxygen, C = carbon; if the names of two elements begin with the same letter, another small letter is attached to the less important, *e. g.* Cl = chlorine, Cu = copper (cuprum). These symbols, however, represent something more than the mere names of the elements; they also convey the idea of a certain definite quantity of each—a quantity which varies in weight for the different elements.

Atoms and Molecules.—This definite quantity is called an *atom*,* and is the smallest amount of an element which can exist in combination with other elements. The smallest quantity of a compound or of an element which can exist in the *free* or *uncombined* state is called a *molecule*;† in the case of the elements a molecule usually consists of two atoms, we therefore write O_2 or 2O for free oxygen, not O;

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* From *a*, not, and *temno*, I cut; *i. e.* impossible of division.

† Diminutive of *mole*, a mass.

Cl_2 or 2Cl for chlorine, &c. As stated above, the atoms of elements vary in weight; the number representing the weight of an atom of any element as compared with hydrogen (the lightest known element) is called its *atomic weight*; *e. g.* hydrogen = 1, carbon = 12.

The following is a list of the more important elements, with symbols and atomic weights:

Name of element.	Symbol.	Atomic weight.	Name of element.	Symbol.	Atomic weight.
Hydrogen	H	1	Cobalt	Co	58·8
Lithium	L	7	Nickel	Ni	58·8
Boron	B	11	Copper (Cuprum)	Cu	63·5
Carbon	C	12	Zinc	Zn	65
Nitrogen	N	14	Arsenic	As	75
Oxygen	O	16	Bromine	Br	80
Fluorine	F	19	Strontium	Sr	87·5
Sodium (Natrium)	Na	23	Silver (Argentum)	Ag	108
Magnesium	Mg	24	Cadmium	Cd	112
Aluminium	Al	27·5	Tin (Stannum)	Sn	118
Silicon	Si	28	Antimony (Stibium)	Sb	122
Phosphorus	P	31	Iodine	I	127
Sulphur	S	32	Barium	Ba	137
Chlorine	Cl	35·5	Platinum	Pt	195
Potassium (Kalium)	K	39	Gold (Aurum)	Au	196·5
Calcium	Ca	40	Mercury (Hydrargyrum)	Hg	200
Chromium	Cr	52·5	Lead (Plumbum)	Pb	207
Manganese	Mn	55	Bismuth	Bi	209
Iron (Ferrum)	Fe	56			

The molecule being commonly considered as two atoms, the *molecular weight* of an element must be twice its atomic weight;* the molecular weight of a compound is the sum of the atomic weights of the elements composing it, *e. g.* water = H_2O; molecular weight $(1 \times 2) + 16 = 18$. Sulphuric acid = H_2SO_4; $(1 \times 2) + 32 + (16 \times 4) = 98$.

Chemical Compounds and Mechanical Mixtures.—We have defined a compound as consisting of two or more elements; we must now direct attention to the essential difference between a *chemical compound* and a *mechanical mixture*. The gases oxygen and hydrogen may be mixed under ordinary conditions in any proportion without the properties of either being lost; the oxygen remains as oxygen and the hydrogen as hydrogen, and by suitable means it is quite easy to demonstrate the presence of each in its natural condition. If, however, two measures of hydrogen be mixed with one measure of oxygen in a closed vessel, and an electric spark be passed through the mixture, *chemical* union immediately occurs, accompanied by some violence, the result being two measures of steam which quickly condenses into drops of water upon the sides of the vessel. The two *gases* have combined, forming a *liquid*, which is water, and which may be proved to consist of hydrogen and oxygen; but the water itself possesses none of the properties of either element. This is a striking instance of a chemical compound produced from a mechanical mixture. We may therefore define a *mechanical mixture* as a body consisting of two or more simpler substances, each of which retains its own individual properties; whereas a *chemical compound* consists of two or more simpler substances, the properties of which are greatly altered.

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* There are exceptions to this rule, viz. phosphorus, the molecule of which consists of four atoms; and mercury and cadmium, which contain only *one* atom.

Laws of Chemical Combination.—The force by virtue of which chemical change occurs is known as *chemical action.*

Chemical action does not proceed in a hap-hazard fashion, it follows certain definite laws. It has been found that when substances unite to form definite chemical compounds, they always do so in the same definite proportions. It is clear that a mere mixture may be made in any proportions the operator may wish; but chemical force does not allow him so to regulate the proportions of the elements in chemical compounds. For example, when sulphur is burned in oxygen a compound known as sulphur dioxide is formed, every atom of sulphur combining with two atoms of oxygen; that is to say, every 32 parts by weight (grains, ounces, &c.) of sulphur combine with twice $16=32$ parts by weight (grains, ounces, &c.) of oxygen, producing 64 parts by weight of sulphur dioxide. Under the circumstances named oxygen and sulphur only combine in this proportion; consequently, if, for example, 36 parts of sulphur were used instead of 32, the additional 4 parts would remain unacted upon *as sulphur*; or, on the other hand, if say 50 parts of oxygen were used, the 18 parts in excess would still remain as *free oxygen.* In fact, sulphur dioxide cannot by any possibility be made to contain even a fractional part more oxygen or sulphur than the above proportions.

Law 1.—Dalton has thus enunciated this law:—"*A definite chemical compound always contains the same elements in the same proportions.*"

Moreover Dalton found that when an element combines chemically with another element to produce more than one compound, the proportional quantities of the elements in these compounds are related in a very simple manner; *e. g.* carbon when burned in a limited supply of oxygen produces a body known as carbonic oxide, which contains carbon and oxygen in the proportion of 12 of carbon to 16 of oxygen; when however, the supply of oxygen is plentiful, another compound, viz. carbon dioxide, is produced, which contains just double that amount of oxygen, 32 parts. No compounds of carbon and oxygen are known which contain less oxygen than carbonic oxide, or more than carbon dioxide, nor are there any containing any intermediate proportions.

Law 2.—The law is expressed as follows:—"*When two elements unite to produce more than one compound, the weights of the constituent elements in these compounds bear some simple relation to each other.*"

Law 3.—Again, *when an element combines with two or more elements to produce separate compounds, the proportion in which it combines with each is either the same or some simple multiple of the same.* As instanced above, 32 parts of oxygen combine with 32 parts of sulphur, and 32 parts of oxygen also combine with 12 or 24 parts of carbon; moreover, when sulphur and carbon unite they do so in the proportion of 12 of carbon to $32 \times 2 = 64$ of sulphur. In fact, whatever be the element combined with carbon, oxygen, and sulphur, the proportions in which these three elements exist will always be either 12, 16, and 32, respectively, or some simple multiple or sub-multiple of those numbers.

Equivalence.—One element may replace another in a compound; thus when zinc is added to hydrochloric acid the hydrogen is eliminated, its place being taken by zinc. These replacements follow the law of definite proportions, but it does not always follow that one atom of hydrogen is replaced by *one* atom of another element. The weight of an element which replaces one atomic weight of hydrogen is known as its *equivalent weight;* in some cases this is the same as the atomic weight, but in many cases (*e. g.* that cited above) the proportion is different. This indicates that the *combining power* of the atoms of different elements varies; one atom of zinc is capable of displacing two atoms of hydrogen, consequently the equivalent weight of zinc is only half the atomic weight, viz. 32·5.

This combining power of an element is known as its *quantivalence* or *equivalence,* or *atomicity,* and is referred to hydrogen as unity. If one atom of an element can replace but one atom of hydrogen it is called a *monad,* and is said to be *monatomic* or *univalent, e. g.* potassium, silver; if two atoms, a *dyad, diatomic* or *bivalent, e. g.* zinc, oxygen; if three atoms, a *triad, triatomic* or *trivalent,* as gold, boron; if four atoms, a *tetrad, tetratomic* or *quadrivalent,* as carbon and tin; if five atoms, a *pentad, pentatomic* or *quinquivalent,* as nitrogen and phosphorus; if six atoms, a *hexad, hexatomic* or *sexivalent,* as sulphur and iron.

Formulæ.—We have seen that elements are expressed by symbols; compounds are similarly expressed by placing the symbols of the constituent elements in juxtaposition. Thus HCl does not mean hydrogen and chlorine, but a compound of these elements known as hydrochloric acid. Such an expression is called a *formula.* If we wish to express the presence of more than one atom of an element in a compound, this is done by adding a small numeral just below and a little to the right of the symbol of that element, thus: H_2SO_4. This shows that sulphuric acid, the compound so represented, consists of two atoms of H, one atom of S, and four atoms of O: these small numerals only multiply the single element immediately preceding them. If we wish to show that two or more molecules of a compound are intended, a large numeral is placed immediately before the formula; thus 3HCl means three molecules of hydrochloric acid, not three atoms of hydrogen and one of chlorine: the large figure so placed multiplies all the symbols as far as the next comma, +, or other sign. The same object is sometimes attained by placing the formula in brackets and adding a numeral lower down to the right, thus: $(HCl)_3$.

Equations.—In order to express chemical change (or *reaction*) an *equation* is employed—that is, the formulæ of the substances entering into the reaction are put down side by side and connected with the sign + (addition); then follows the sign = (equivalent to), followed by the results of the action, thus:

$$4HCl + Zn_2 = 2ZnCl_2 + 2H_2;$$
$$K_2 + 2H_2O = 2KHO + H_2.$$

It is needless to point out that the number of atoms of each element must be the same on both sides of the sign =.

Formulæ and equations of this character are sufficiently expressive for the comparatively simple compounds and changes which occur in inorganic chemistry, but in organic chemistry, *i. e.* the division of the science which treats of the products of living organisms or artificially produced compounds allied to these, it is necessary to modify these formulæ so as to express the real or supposed manner in which such compounds are built up from simpler compounds. To express organic compounds by formulæ giving simply the number of atoms of each element would be completely unintelligible, especially as there are numberless compounds, differing more or less widely in physical and chemical characters, which are composed of the same elements united in exactly the same proportions.

To meet this difficulty compounds are viewed as being composed of *radicals*.

Radicals.—Radicals are either elementary, as Cl^{i}, O^{ii}, C^{iv}, &c., the small Roman numerals representing the number of atoms of hydrogen required to satisfy their chemical affinities; or compound, which are compounds having one or more of their affinities unsaturated, *e. g.* carbon forms the saturated compound *methane*, CH_4, the removal of one atom of hydrogen from which gives CH^{i}_3, *methyl*, a compound radical. This compound radical is capable of combining with a single atom of a monad element or other monad radical forming saturated compounds—CH_3I,

CH_3OH, CH_3OCH_3 or $(CH_3)_2O$, &c., the radical CH_3 being here analogous to an element such as K, which forms the corresponding compounds KI, KOH, K_2O (or KOK).

Such a method of expression reduces the study of organic chemistry from a chaos to something like order; the more complex the compound, the more clearly is this seen. This explanation is given in order to account for the use of such formulæ in equations in this work, but the student must familiarise himself with this subject by the study of some elementary work on Organic Chemistry.

Nomenclature.—It is now necessary to explain the method adopted for naming chemical compounds, or *chemical nomenclature.* Compounds consisting only of oxygen and one other element are called *oxides;* similarly we have chlorides, iodides, bromides, fluorides, sulphides, phosphides, &c. In many cases the oxides readily combine with water, producing substances called *acids,* having very marked properties—a sour taste, the power of reddening blue litmus and many other vegetable colours, and especially the property of combining with the metallic elements or their oxides, with production of compounds in which the *acid* action on colours is wholly or partially destroyed, and hydrogen is eliminated from the acid, it being replaced by the metal: oxides capable of thus forming acids are called *anhydrides.* The metallic oxides, as a rule, neutralise the acids, and are called *bases* or *alkalies.* It frequently happens that an element may produce several distinct compounds with another element; for instance, nitrogen forms five oxides. To distinguish such compounds certain prefixes are commonly employed to determine the number of atoms of oxygen in the molecule, *mono-, di-, tri-, tetra-, penta-,* &c., the oxides of nitrogen being as follows:—Nitrogen monoxide, N_2O; dioxide or binoxide, N_2O_2, (2NO); trioxide, N_2O_3; tetroxide, N_2O_4, ($2NO_2$); pentoxide, N_2O_5. The same prefixes are used for other compounds; for instance, we may have monosulphide and disulphide of iron, &c. Another common method, which has much less to recommend it, but was formerly much in vogue, is to add the prefix *sub* or *per* to express the compounds containing a small and a large proportion of an element respectively; *e. g.* subchloride and perchloride of mercury, HgCl and $HgCl_2$. Again, the name of an acid or base containing much oxygen is made to end in *ic*, while one containing less oxygen ends in *ous*; thus nitrous and nitric acids correspond respectively with the *tri-* and *pent*-oxides of nitrogen. If an acid contain less oxygen than the *-ous* acid the prefix *hypo* is adopted, as in hyposulphurous and hypophosphorous acids; whilst one containing more than the *ic* acid may be called *per*-chloric, &c., or sometimes *hyper*, or even *super*. When an acid combines with a base the compound is called a *salt*, the salts of an *-ous* acid being made to end in *ite*, and of an *-ic* acid in *ate*, *e. g.* sulph*ite* and sulph*ate* of sodium. If the acid has been completely saturated with the base, so that no more hydrogen can be displaced, the salt is called a *neutral salt*, and is commonly neutral in its action upon litmus. Sometimes, however, only a portion of the hydrogen is removed, the salts being then called *acid* salts, such as bitartrate of potassium; and sometimes again an excess of base may be dissolved, producing *basic* salts, *e. g.* subacetate of lead.

Questions on Chapter I.

1. What is an element? Name twelve elements, with symbols and atomic weights.
2. What is a molecule? How would you find the molecular weight of a compound having the composition H_3PO_4?
3. Enunciate the three laws of chemical combination.
4. What is meant by quantivalence? Name one element which is monatomic, one diatomic, &c., up to hexatomic.
5. Explain the use of formulæ and equations.
6. What is the significance of each of the following prefixes and affixes?—per, sub, hypo; ous, ic, ite, ate, ide.
7. What are acids, anhydrides, bases, salts?

The above questions should be answered by the student in *writing;* nothing tends to fix facts in the memory more effectually than the constant habit of committing them to paper.

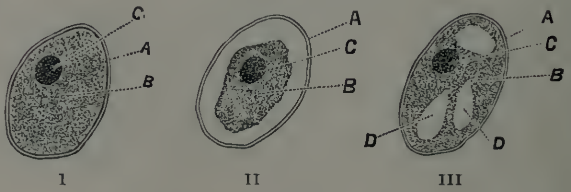
CHAPTER II

STRUCTURE OF PLANTS

Before drugs can be used for the production of preparations it is manifestly necessary that they should be brought into a suitable state for working up; this usually entails the operations of *collection*, *drying*, and *comminution*. The majority of crude drugs being of vegetable origin, and comparatively few of these being indigenous (native) to this country, the operations of collection and drying are already performed by the time they reach the British pharmacist; but as there are many medicinal plants growing either wild or in cultivation, it will be necessary to give a very short description of the parts of the plants employed and their structure, together with the time of the year most suitable for their collection, and the means of drying; moreover drugs as imported usually require further drying before they can be reduced to powder.

Cells and their Contents.—All parts of plants are composed of various modifications of the *cell*, which in its simplest complete form is a more or less rounded body, consisting of three distinct parts, as shown in Fig. 1: A, the *cell-wall*, a thin transparent skin; within which is a semi-fluid, somewhat granular substance, called *protoplasm* (B); and C, the *nucleus*, which is a rounded denser portion, lying in the

Fig. 1.



protoplasm, usually nearer one side of the cell. While young the protoplasm completely fills the cell cavity, but as growth proceeds hollow spaces or *vacuoles* (D, Fig. 1, III) make their appearance, which are filled with a clear watery liquid called *sap*. If a fully grown cell containing sap be immersed in alcohol for a few seconds, and then examined microscopically, it will be seen that the protoplasm has contracted from the cell-wall owing to the solution of the sap in the alcohol, the cell presenting the appearance shown in Fig. 1, II. The cell-wall permits of the ready transference of water and many dissolved solids through itself, consequently the sap can readily pass from cell to cell, carrying with it such substances as sugar.

Other substances are frequently found in cells; the most important for our consideration are chlorophyll, starch, lignin, and raphides.

Chlorophyll is the green colouring matter of leaves, some stems, &c., and usually occurs in granules floating in the sap of the cells immediately beneath the surface of the leaf.

Starch is most commonly found in the older parts of plants, such as roots, and stems, and in seeds; familiar examples of drugs containing large quantities of starch are ipecacuanha root and ginger. It occurs as minute granules, usually quite distinct from each other, but varying much in size and form, according to the plant from which it is obtained, and is commonly seen floating in the sap. Starch is unaffected by cold water, but, as is known to all, when boiled with water it swells up, producing a "jelly" or "pudding," according to the quantity present. This fact often renders starch troublesome to the pharmacist.

Lignin.—This substance is not found floating in the sap, but is produced as a kind of inner coating on the cell-wall; it is always present in the harder parts of plants, such as wood, much of the bark, the stones of fruits, &c. In composition it much resembles the cell-wall itself. It gives strength and solidity to the parts in which it occurs.

Raphides are minute crystals, which occur either singly or in groups in the cells of many classes of plants, and usually consist of oxalate of calcium; they are abundant in rhubarb.

The cell varies much in form under different conditions: when growth is free and nutriment abundant it is usually spherical or nearly so; when the pressure of surrounding cells is equal or nearly so on all sides they become more or less regularly polygonal. When nutriment is chiefly derived from one direction only, and especially if the pressure of surrounding cells is in a different direction, cells having an elongated form are produced. These last are almost invariably much thickened by deposition of lignin; in fact, when very old the cell cavities are frequently almost entirely filled up.

The combination of cells produces a *tissue;* four kinds are distinguished:

1. *Parenchyma.*—This consists of cells whose length does not greatly exceed their breadth, and whose walls are not much thickened. This kind of tissue forms the great bulk of soft plants, and of the leaves and pith, and of parts of the stem, fruit, &c., of many trees, especially palms.

2. *Prosenchyma.*—This tissue consists of long narrow cells, usually much thickened, tapering at each end, and consequently overlapping one another. The wood, bark, veins of leaves, &c., are composed of this tissue. There is a considerable difference between the prosenchyma of wood and bark, the latter consisting of longer, very much thickened cells, of a tougher and more flexible nature.

3. *Vessels or Vascular Tissue.*—Vessels are in reality nothing but a combination of several cells, the divisions between which have been removed by absorption. They occur in wood, bark, &c,, usually in combination with prosenchyma. They are of various forms.

4. *Epidermal Tissue or Epidermis.*—In most plants the outer surface consists of cells of different shape from those of the other parts of the plants, forming a firm layer, which is usually easily separated from the tissue beneath, and is called the epidermis. The cells are commonly of a more or less flattened tabular character.

Parts of the Plant.—From these simple "elements" all the various organs of the plant are built up. We will now briefly describe these parts.

When the seed of a flowering plant germinates growth takes place in two directions—downwards into the earth, and upwards into light and air. These parts are called respectively the *root* and *stem*.

The Stem.—The young stem usually carries upwards one or more green leaf-like appendages, but these quickly disappear, true leaves being formed. The leaves are not produced hap-hazard upon the stem, but at regular points called *nodes*, and each leaf commonly bears in its *axil* (*i. e.* angle between the leaf-stalk and upper part of stem) a bud, which as it grows produces a young stem called a *branch*. The stem most commonly continues to develop in a more or less vertical direction, bearing branches, which in turn bear younger branches and *twigs*, and these produce leaves. Frequently, however, the stem runs along either upon the surface of the ground, or in many cases beneath the earth. That these creeping or underground stems are not roots is proved by the fact that either at regular intervals (nodes) or upon their entire course buds are produced upon their upper surface, from which young stems arise into the light, producing leaves, the under side throwing off slender rootlets. Roots never produce buds or leaves. There are many examples of more or less underground stems amongst the drugs of the Pharmacopœia, the term *rhizome* being applied to the majority of such stems, *e. g.* podophyllum, ginger, and valerian; these should be examined by the student, the first especially being very instructive, the scars where rootlets, &c., have broken off being very evident. Another form of underground stem is seen in the mint and the rose, which is called a *sucker;* it is more slender than a rhizome, and after sending off many rootlets and leafy stems, finally itself turns up into the air.

Other underground stems take the form of more or less rounded enlargements at or below the surface of the ground, leaves arising from above and rootlets from below. These enlargements are of several kinds, the most common being *corms* and *bulbs*. A *corm* consists of a central solid portion surrounded by one or several thin membranous scales, really modified leaves; colchicum and saffron are examples. A *bulb* consists of numerous thick fleshy scales enclosing one another in a concentric manner, either covered externally by thin scales or not; the squill is a good example, and, although not pharmaceutical, the onion.

Very frequently the stem is practically nothing more than a mere stump, the leaves arising directly from the surface of the earth, as in the dandelion.

The aërial stems of plants vary in their duration of life: some die down annually, as, *e. g.*, the *mints* and *henbane;* these are called *herbs:* while others live for several years, the same stem producing leaves each season; these are called *trees* or *shrubs,* according to their size and general appearance: the *oak* and *rose* are familiar examples.

Structure of Stems.—If the stem of one of our common trees be cut across it will at once be seen that it is not uniform in structure, three main divisions immediately become apparent: the central portion is of soft tissue, and is called the *pith* or *medulla;* outside this are numerous rings of hardened tissue forming the *wood,* which in turn is surrounded by a ring of tissue which is commonly softer than the wood, but tough, called the *bark.*

The pith is composed of parenchymatous tissue, and it will be seen that lines of similar tissue radiate from the pith to the bark; these are called *medullary rays.*

The wood is not uniform; it consists of prosenchyma and vascular tissue, and presents a considerable difference in appearance according to the rate of growth, hence the "rings" which indicate each year's growth.

It.may frequently be seen that the wood near the centre is of much darker colour and firmer than that near the circumference; this is due to the deposition of matter (resinous and otherwise) in the cells of the older and more central wood. This dark wood is known as *heart-wood* or *duramen,* the softer and paler portion being called *alburnum* or *sapwood.* Guaiacum wood is a good example of duramen.

The bark which forms the outer portion of the stem is composed of three layers, called respectively *inner, middle,* and *outer* bark—*endo-, meso-,* and *epi-phlœum.* The inner bark is composed of a variety of woody cells called *liber* or *bast* cells, mixed with some parenchyma and *laticiferous* vessels; these latter are so called because they serve as carriers of *latex,* a kind of milky juice; they are branched vessels. The middle layer is commonly coloured green; it is built up of loose parenchyma, with many spaces between the cells, and a few laticiferous vessels. The outer layer, sometimes largely developed, as in the cork oak, is composed of tabular cells.

The arrangement of the parts of the stem of a palm or tree fern is different from that of our common trees, but it is not necessary to describe them here; the student is referred to a work on botany.

The Root.—The young root very frequently continues to grow downwards into the earth as one main root, from which branches and rootlets are developed at irregular intervals. Such a root is called a *tap-root;* it is well seen in the horse-radish, dandelion, and aconite. Different terms are applied to distinguish variations of form and character. Very frequently, however, there is no evident main root, but the appearance is rather that of a bunch of fibres, as in many *grasses*, and the rootlets springing from the corm of *colchicum*, &c.; such roots are called *fibrous*.

Roots increase in diameter by the formation of annual layers of wood tissue, and in length by the growth of new cells just within the apex (*i. e.* opposite extremity to that which joins the stem). There is usually no pith, but if cut across, medullary rays can be discerned, and a bark is evident; this bark is sometimes called the *cortex*, and the inner woody portion the *meditullium*, they are well seen in ipecacuanha, also in dandelion: if the latter root be cut across while fresh the bark appears white, whilst the wood is yellow.

The Leaf.—A leaf in its most complete form consists of three portions: (1) an expanded portion, commonly flattened, called the *blade*, *limb*, or *lamina;* (2) a narrower portion, connecting the lamina with the stem, called the *petiole* or *leaf-stalk;* and (3) a flattened portion at the base of the petiole, either encircling the stem or on either side of the petiole, called the *stipule*. Very frequently one or other of these parts is wanting.

It will be noticed that the lamina is intersected by numerous fibrous ramifications called *veins;* these veins are composed of cells similar to those of the wood and bark, whilst the softer part of the lamina consists of parenchyma, very many of the cells containing chlorophyll.

The parts of the plant which we have so far considered are called the *organs of nutrition,* because their office is to continue the life of the individual plant by absorbing nutriment from the soil and air; we now pass to a very brief mention of the *organs of reproduction,* whose special function it is to provide for the continuance of the species by the production of seed.

The Flower.—The organs of reproduction are the flower and its appendages, from which are produced the fruit containing the seed.

Flowers are placed either at the extremity of a branch, or they arise from the axil of a leaf, which in this case is called a *bract* or *floral leaf;* the bract is usually green like the ordinary leaves, but frequently coloured. The stalk upon which a single flower is borne is called the *peduncle;* if, however, the floral axis branches, and each branch bears a flower, the main axis is still called a *peduncle,* and the branches are called *pedicels.* The varieties of arrangement of the flowers upon the axis (or inflorescence) give rise to different terms to distinguish them, but we are only concerned with one broad distinction into *indefinite* and *definite.* In the varieties of *indefinite* inflorescence the flower-buds in the lower part of the axis open first, to be succeeded by those next above them, the axis continuing to elongate; and as its apex is never terminated by a flower-bud, the growth continues as long as the season and other surroundings permit. In the *definite* inflorescence, however, the apex of the axis produces a flower-bud which of necessity terminates its growth; this bud is the first to expand, the other flowers following in regular progression downwards. When the inflorescence is flattened, as in the elder and hemlock, definite inflorescence proceeds from the centre to the circumference (*centrifugal*), indefinite from the circumference to the centre (*centripetal*). Very frequently the inflorescence consists mainly of bracts, as in the *strobiles* of the hop, which are formed of densely packed membranous bracts, with inconspicuous flowers or seeds.

Several drugs of the Pharmacopœia are directed to be collected when about one third of the flowers have expanded, *e. g.* aconite; this will be at about the time when the oldest flowers are producing fruit.

The student should now obtain some flowers of the common buttercup, in various stages of growth. He will observe that the parts of the flower are arranged in rings or *whorls* upon the apex of the peduncle. These whorls, proceeding from without inwards, are as follows :—Taking a flower which is just expanding from the bud, he will notice *five* small separate outer leaves, coloured yellowish, and either spreading or sharply curved back (these fall off soon after the flower opens) ; they are called *sepals,* and together constitute the *calyx.* Immediately within the calyx are five larger bright yellow leaves, also separate; they are called *petals,* and form the *corolla.* Having stripped off both calyx and corolla, he will find a large number of stalk-like parts, each bearing a thickened oblong head; the stalk is called the *filament,* and the head the *anther,* the two forming a *stamen,* the stamens altogether being called the *andrœcium.* Within the stamens, and forming the centre of the flower, are a number of small bodies closely packed together; if one of these be removed and cut through lengthwise, it will be seen by the help of a lens to consist of a somewhat thickened hollow portion below, containing a single pale-coloured rudimentary seed; the upper part is a short stalk, the apex of which is somewhat like a saw in outline. The lower hollow portion is the *ovary,* enclosing the *ovule;* the upper stalk is the *style,* surmounted by the saw-like *stigma.* In many plants the style is very long—as, for instance, in the crocus. The whole organ is called a *carpel,* the carpels together forming the *gynœcium,* or *pistil.*

Several drugs consist of the whole or parts of flowers, *e. g.* elder flowers, rose and red poppy petals, saffron—the stigmas and styles of *Crocus sativus.*

The andrœcium and gynœcium are the *essential* organs of reproduction, they alone being necessary for the formation of fruit and seed; the corolla and calyx serving as protection for these delicate organs, and by their colour and odour attracting insects, which often effect the fertilisation of the ovule.

The Seed.—For a detailed description of the mode of production of the seed the student must consult some work on botany; it will be sufficient here to mention that the anthers contain a fine dust called *pollen*, which is shed upon the stigma, and by development fertilises the ovule, which finally produces the seed. The seed is enclosed in the fruit, which varies much in character in different plants: it may be either soft and juicy, as in the plum and grape, or hard and dry, as in the poppy head and nut; it may contain one or many seeds; may open and discharge the seeds, or it may remain till the outer portion rots away and so exposes the seed. Many fruits and seeds are used in medicine, and parts of others—as, for example, the outer portion of the peel of the lemon and orange, the juice of the lemon, mulberry, and buckthorn, the seeds of stramonium and mustard.

Questions on Chapter II.

1. Describe a complete cell.

2. Where does starch usually occur? What is its behaviour towards cold water and boiling water respectively?

3. What is lignin?

4. Distinguish between parenchyma, prosenchyma, and vascular tissue. Describe a woody cell.

5. What is a rhizome? Give six examples from B. P.

6. Distinguish between a corm and a bulb.

7. Describe the parts of the stem of the oak. What is duramen?

8. How does a root differ from a stem?

9. What are the parts of a leaf?

10. Describe the flower of a buttercup.

11. What is an indefinite inflorescence?

12. Define bract, lamina, tap-root, liber, epiphlœum, epidermis, chlorophyll.

13. Give official examples of the following parts of plants used as drugs:—Petals, duramen, root, stigma, corm.

CHAPTER III

COLLECTION, DRYING, AND COMMINUTION

Having briefly described the parts of the plant and their structure, we will now give a table of indigenous plants, with the parts employed and time of year for their collection; after which the method of drying will be considered.

Name of plant.	Part used.	Time for collection.
Aconitum napellus†‡ (cultivated)	Dried root	Early spring—B. P.
	Fresh leaves and flowering tops	When one third of the flowers have expanded; June or early July.
Peucedanum graveolens* (cultivated)	Dried fruit	End of July and August.
Anthemis nobilis* (cultivated or wild)	Flower-heads	August and September.
Cochlearia armoracia (cultivated)	Fresh root	Autumn and early spring, before leaves appear.
Atropa belladonna‡ (wild or cultivated)	Dried root	Autumn or early spring.
	Dried leaves Fresh leaves and young branches	When fruit begins to form; late in June, July, and early in August.
Carum carui (cultivated)	Dried fruit	August.
Colchicum autumnale§ (wild)	Fresh corm	From end of June to end of September.
	Corm without coats, dried	—
	Dried ripe seeds	June and July.
Conium maculatum‡ (wild)	Fresh leaves and young branches	When fruit begins to form; June and July.
	Dried fruit	When fully *grown* but still *green;* July and August.
Coriandrum sativum* (cultivated)	Dried ripe fruit	End of July and August.

* Very little now cultivated in this country.

† Mr. P. W. Squire has shown that the roots are most active in autumn (P. J., 1889, p. 645).

‡ When the flowering has well advanced, but before the seeds ripen, the leaves are considered to be most active.

§ Opinions differ as to the proper time for collecting colchicum corms; some recommend June and July, others August and September.

Name of plant.	Part used.	Time for collection.
Digitalis purpurea† (wild)	Dried leaves of plants in second year	When about two thirds of flowers have expanded; July and early in August
Ecballium elaterium (cultivated)	Nearly ripe fruit	Late in July and August.
Aspidium filix-mas (wild)	Dried rhizome, divested of scales, roots, and dead portions	Late in autumn.
Glycyrrhiza glabra (cultivated)	Fresh and dried subterranean stems and roots	Autumn.
Hordeum distichon (cultivated)	Dried seed, divested of its coats	August and September.
Triticum vulgare (cultivated)	Dried and powdered seed	August and September.
Hyoscyamus niger† (cultivated or wild)	Fresh leaves, flowers, and small branches, also dried leaves and flowering tops, from plants in second year	When about two thirds of flowers have expanded, *i.e.* end of May till early in July.
Lactuca virosa† (wild or cultivated)	The whole herb	While flowering; late in June and July.
Pinus larix‡	Dried inner bark	Spring.
Prunus laurocerasus	Fresh leaves	Late autumn.
Linum usitatissimum* (cultivated)	Dried ripe seeds	September.
Humulus lupulus (cultivated)	Dried strobiles (fruits with bracts).	Late in August and September.
	Glands from the strobiles	—
Daphne mezereum Daphne laureola	Dried bark of root or stem	October.
Morus nigra (cultivated)	Ripe fruit (juice of)	End of August and September.
Juniperus communis	Fresh fruit (oil from)	Full-grown but unripe and green; October.
Lavandula vera (cultivated)	Fresh flowers (oil from)	August.
Mentha piperita (cultivated)	Fresh herb (oil from)	While flowering; July and August.
Mentha viridis (cultivated)	Fresh herb (oil from)	While flowering; July and August.
Pinus sylvestris	Fresh leaves (oil from)	Autumn.
	Wood (tar from)	—
Rosmarinus officinalis (cultivated)	Flowering tops (oil from)	End of August and early in September.
Ruta graveolens (cultivated)	Fresh herb (oil from)	June and July.
Papaver somniferum (cultivated)	Dried fruits	When nearly ripe; July and August.
Quercus robur‡ (wild)	Dried bark of small branches and young stems	In spring and in June.

* Very little now cultivated in this country.

† When the flowering has well advanced, but before the seeds ripen, the leaves are considered to be most active.

‡ In spring the bark separates from the wood most easily.

Name of plant.	Part used.	Time for collection.
Papaver rhœas (wild)	Fresh petals	June and July.
Rhamnus catharticus	Fruit	September.
Rosa canina, &c. (wild)	Ripe fruit	September.
Rosa centifolia (cultivated)	Fresh petals	When fully expanded; June and July.
Rosa gallica (cultivated)	Fresh and dried petals	When unexpanded; June and early July.
Juniperus sabina (cultivated)	Fresh and dried tops of branches	In early spring.
Sambucus nigra (wild)	Fresh flowers	June and July.
Cytisus scoparius (wild)	Fresh and dried tops of branches	End of May and early June.
Brassica alba (cultivated)	Dried seeds	When ripe; August and early September.
Brassica nigra (cultivated)	Dried seeds	When ripe; August.
Solanum dulcamara	Younger stems	September and October.
Datura stramonium (wild)	Dried leaves	July.
	Dried seeds	When ripe; September.
Taraxacum officinale (wild)	Fresh and dried root	Autumn—B. P. (it is more bitter in spring).
Arctostaphylos uva-ursi (wild)	Dried leaves	September and October.
Valeriana officinalis (wild or cultivated)	Dried rhizome and rootlets	Autumn.

The tools required for the collection of plants are obviously the common garden implements.

Drying.—The drug having been collected, it has now to be subjected to the operation of *drying* or *desiccation*. The one principle to be borne in mind is that, for successful drying, *the operation must be performed as quickly as possible, but with the least exposure to heat.* By successful drying is meant the removal of moisture without effecting changes in the other constituents of the drug. For this purpose the drug is usually placed in a specially constructed drying cupboard or room, the temperature of which can be regulated; such cupboard may be heated by gas, or, as is more usual in large laboratories, by means of steam. In order to attain the maximum drying effect from a given heat, it is necessary that there should be free circulation of air through the closet, and the drug should be arranged in thin layers, which should be frequently turned over. The accompanying diagrams illustrate a form of drying closet which is due to Mr T. E. Greenish.

Fig. 2 is a front view of the drying closet with the door open. Fig. 3 is a transverse vertical section of the same, taken in the line 1, 2 of Fig. 2.

Fig. 3.

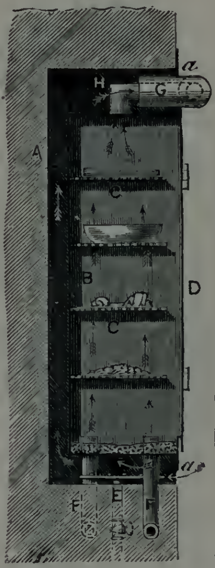
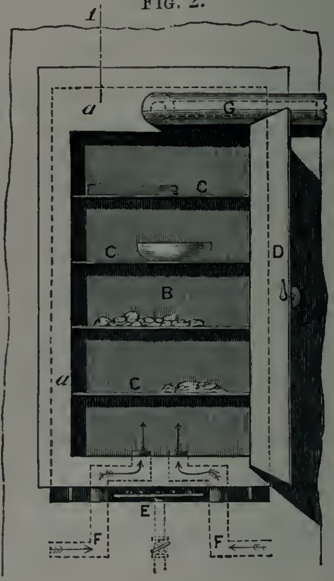


Fig. 2.



A is a recess in the wall; B is the drying closet, made of thin sheet iron, and provided with wire shelves, C, and a door, D. This closet is made of such a size that when fixed into the recess in the wall a space of about two inches is left at the back and sides, and also at the top—in fact, all round the closet, the space being covered in front by the flange *a a*, formed in the front of the closet. E is a gas burner, and F F are two air-pipes, which enter the bottom of the closet B; these pipes draw their supply from the air *external* to the laboratory, and their upper ends are surrounded by a layer of sand forming a sand-bath two inches deep. The gas being lighted is supplied with air from the front, and the heated air, together with the products of combustion, pass round the closet through openings made for that purpose in the sides and back of the gas chamber, up the space between the closet and the wall, to a pipe G, and thence to a chimney.

The articles to be dried or evaporated are placed either upon the shelves in the closet, or upon the sand-bath at its bottom, according to the degree of heat that may be thought desirable. The air which enters by the pipes F F, slightly warmed by the sand, will carry up any vapour therefrom to a pipe, H, at the top of the closet, and thence to the pipe G. In order to regulate the draught of air at the back and sides of the closet, and thereby to adjust the degree of heat, the pipe G is provided with a circular damper, and the gas chamber also has in front of it an arrangement for regulating the supply of air to the gas, thus preventing sudden fluctuations of temperature. By these means the heat of the closet may be adjusted to the greatest nicety.

It will be observed that the closet is heated by gas, which, as a source of heat for this purpose, possesses advantages over any other. It is always ready to hand, continuous, and can be regulated to any required temperature.

The gas, being lighted, heats the sand-bath immediately over the jets; part of the heat, passing through the openings in either side and back of the gas chamber, fills the space all round between the closet and wall, the products of combustion escaping by the flue G into the chimney, cannot by any possibility come into contact with the contents of the closet.

A draught of fresh air is provided for by the two air-tubes, F F, which derive their supply from the atmosphere *outside* the building, and have no connection with the laboratory, so that when the air-tight door of the closet is closed its contents are perfectly protected from contamination by noxious vapours, &c., whilst the drying process is in operation. These tubes pass through the gas chamber, from which they derive some heat, and as soon as they enter the sand bend at right angles, and for six inches or so traverse the hot sand; another bend upwards enables them to deliver their supply of air fresh, and to a certain extent warmed in its passage through about ten inches of heated tube.

The moisture from the material in the course of being dried is carried off by the tube H at the top of the closet; this tube enters the gas chimney, and is carried for some distance through its interior. By this arrangement the heat of the chamber external to the closet, on its passage into the chimney, warms the interior tube, and thus contributes to the required draught.

The shelves are made of wire, in the form of a shallow dish, and duplicates of sheet iron are provided to take their place when desired. The temperature of each shelf differs, the lowest being the hottest.

The closet may be maintained, with a very trifling variation, at the same temperature for any length of time, day and night, without requiring special attention, and the constant supply of fresh air, warmed as it enters, necessarily facilitates the drying process.

Various modifications may be applied to the drying closet. A very important one is to have the inlet of air at the top and the exit at the bottom. The advantage claimed for this arrangement is that the heated air becomes cooled by the evaporation of moisture from the drugs, and therefore heavier, consequently assisting the downward draught. It is, however, more difficult of arrangement. The air which supplies a fireplace possessing a good flue should be drawn downwards through a drying closet, before entering which it should be warmed by traversing a flue or pipe heated by the fire.

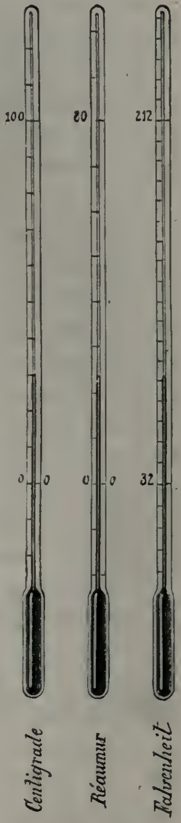
Other forms may readily be arranged, by which the flue of an ordinary stove is utilised as the source of heat.

The reason why it is necessary to ensure a good draught through the cupboard is very simple. Air becomes readily charged with aqueous vapour up to a certain degree, the *proportion* of aqueous vapour being *constant* for each degree of temperature; the higher the temperature, the greater the amount of moisture taken up by the air. If, therefore, the air of the drying closet were not changed, it would soon be completely charged with moisture, after which no desiccation would be possible; with a good draught, however, the moist air becomes replaced by warm dry air, which can in turn take up more moisture, and is carried out by the exit.

Thermometers.—The temperature of the closet is registered by means of thermometers, of which it is advisable to have two—one near the top and one near the bottom.

For a general description of thermometers, their construction, &c., the student is referred to a good text-book of physics, such as Deschanel or Ganot.* It is sufficient to state here that their use depends upon the fact that liquids expand as the temperature rises, and contract as it falls. Mercury is the liquid commonly employed, on account of its long range of comparatively uniform expansibility. Thermometers are of three kinds; Fahrenheit's, Centigrade or Celsius's, and Réaumur's.

FIG. 4.



The scale of the Centigrade thermometer is divided into 100 equal parts or degrees (written °) between the boiling-point and freezing-point of water, which are marked respectively 100° C. and 0° C.; Réaumur's into 80 degrees, 80° R. and 0° R.; while Fahrenheit's contains 180 degrees between the same constants, marked 212° F. and 32° F. (see Fig. 4). It is evident, therefore, that 10° C. are equal to 8° R. or 18° F. The zero of Fahrenheit's scale was taken at the temperature of a mixture of ice and salt. The rules for the conversion of the different scales are as follows:

To convert C. into R. multiply by 0·8.
,, R. ,, C. divide by 0·8.
,, C. ,, F. multiply by $\frac{9}{5}$ and add 32.
,, F. ,, C. subtract 32 and divide by $\frac{9}{5}$.
,, R. ,, F. multiply by $\frac{9}{4}$ and add 32.
,, F. ,, R. subtract 32 and divide by $\frac{9}{4}$.

The reasons for these formulæ will be evident on consideration.

Thermometers should be kept some months before use, because the bulb undergoes a slight contraction soon after making, with consequent rise of the mercurial column. They should then be tested against a "standard" thermometer, and a record kept of the errors of the scale, which will frequently amount to 2° or even 3° C.

The Centigrade thermometer presents certain advantages over both of the others, and is almost universally employed on the Continent and in this country for scientific purposes, although Fahrenheit's is used here for other purposes. Throughout this work temperatures are given in Centigrade degrees; a table of equivalents of F. and C. scales will be found in the Appendix.

In drying drugs the temperature usually found most satisfactory is from 37° C. to 60° C. Several considerations must be allowed for, the chief of which are the following:

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* See also 'P. J.' (iii), xix, pp. 1011, 1030.

1. If the drying be too slow fermentation is apt to occur, with consequent destruction of the drug by changes of colour and production of acidity; for instance, henbane becomes almost black and sour if not rapidly dried. The temperatures most favorable to fermentation are from 15·5° C. to 37° C.

2. The green colour of fresh leaves is impaired if dried at a temperature exceeding 60° C., or if exposed to a strong light whilst drying.

3. Drugs containing starch must not be dried at a temperature exceeding 71° C., or the starch swells and becomes partially soluble in cold water. Albumen is coagulated at a temperature of about 93° C.

It will generally be found that for green herbs and leaves a temperature near 49° C. is the most suitable, whilst roots and woody substances should be dried at 60° C., unless they contain any special constituent which would be injured at that temperature. Flowers may be dried like leaves, unless they contain an odorous principle, in which case a temperature of 37° C. is more suitable.

Rate of Drying.—The rate at which drugs lose their moisture increases rapidly as the temperature rises; *e. g.* if a given quantity of drug can be dried in four hours at 65·5° C., it will take about sixteen hours at 37·7° C., other conditions being the same. This subject will be again referred to in a subsequent section.

The rapidity of desiccation also depends to a great extent upon the nature of the drug. Leaves and substances of a light or porous nature dry much more quickly than denser and thicker drugs, like roots, on account of the immensely greater surface exposed to the drying influence of the hot air, in proportion to the weight of drug used. It also depends upon the dryness of the surrounding atmosphere. If the amount of moisture present in the air approaches the maximum limit, desiccation of drugs, &c., proceeds very much more slowly than if it be far removed from that point; hence the necessity for a draught of *warm, dry* air. The ordinary air of the laboratory is often very much charged with steam, and therefore unfitted for the drying closet, which should draw its supply from an external source.

Loss in Drying.—The amount of moisture lost by drying different drugs varies greatly. A table of the loss sustained by partial drying and grinding will be found on page 42; the fresh leaves and young tops of plants such as henbane, conium, or belladonna lose about 82 to 88 per cent. by drying, and fresh roots like taraxacum lose about 75 to 80 per cent.

The student should obtain some fresh leaves of belladonna, stramonium, aconite, or flowering tops of henbane, or, failing these, some dandelion leaves, and, having weighed them in the fresh state and recorded the weight in his notebook, they should be spread out on trays in the drying closet at a temperature of 40°—50° C. If no well-constructed drying closet be available, the student may yet gain much by arranging the leaves on a wire tray over a kitchen fireplace, in such a position that the draught passes over or through the tray. The leaves should be turned after about six hours, and when quite crisp to the touch may be removed to a tin or box. After standing in the box for about one day they should be examined, and if still dry to the touch they may be weighed, and the percentage of dry material calculated. It may, however, happen that the leaves will be no longer quite dry, because the midrib and thicker veins were not perfectly dried, and this moisture has diffused itself through the lamina; in this case a few hours' further drying should be given them. If successfully dried they should be distinctly green, and possess a well-marked odour.

This being the first operation in which weighing has been mentioned, it will be convenient to describe in this place the weights and measures commonly employed in pharmaceutical operations.

Metric System.—The simplest system of weights and measures is undoubtedly that known as the "metric system;" it will therefore be first described, although not commonly in use in this country except for analytical operations. It is almost universally employed on the Continent for all trade purposes.

The standard from which all the measures and weights have been derived is the "metre," hence the term "metric" system. This standard is an arbitrary one, and is the length of a forty millionth part of the circumference of the earth around the poles. The other linear measures are as follows :

The *Kilo*metre = 1000 metres.
,, *Hecto*metre = 100 ,,
,, *Deka*metre = 10 ,,
,, Metre
,, *Deci*metre = $\frac{1}{10}$ metre.
,, *Centi*metre = $\frac{1}{100}$,,
,, *Milli*metre = $\frac{1}{1000}$,,

It will be observed that all the above measures greater than the metre are obtained by *multiplication* of the metre by 10 or a multiple of 10 ; whilst all less than the metre are obtained by *division* of the metre by 10 or a multiple of 10 ; also that the Latin prefixes are employed to indicate division, and the Greek multiplication of the standard. These rules apply throughout the whole system of weights and measures, all are related to each other by 10 or multiples of 10 ; hence the name "*decimal*," which is also commonly employed to designate this system. Measures of *surface* are not frequently of use in Pharmacy ; it will suffice to mention that the unit of surface is called the "are." It is the square of ten metres—that is, it contains 100 square metres.

The unit of *capacity* is the "litre," which is the cube of a decimetre ; its multiples and subdivisions are as follows :—The kilolitre, hectolitre, dekalitre, decilitre, centilitre, and millilitre. A litre is sometimes represented as "1000 c.c.," that is cubic centimetres, the c. centimetre being the term usually applied to the millilitre in analysis.

The unit of *weight* is called the "gramme ;" it is the weight of a *cubic centimetre* (or millilitre) of distilled water at a temperature of 4° C. From the gramme (sometimes written "gram") other weights are derived, viz. kilogramme, hectogramme, dekagramme, decigramme, centigramme, and milligramme. It will be noticed that a litre (1000 c.c.) of water at 4° C. weighs 1000 grammes or 1 kilogramme. 4° C. is the temperature employed because that is the point at which water is most dense, *i. e.* a given weight occupies least space.

English Weights and Measures.—We now pass to the consideration of the weights and measures in common use in this country as far as they relate to Pharmacy.

Avoirdupois Weight.

1 grain, gr.
1 ounce, oz. = 437·5 grs.
1 pound, lb. = 16 oz. = 7000 grs.
1 quarter, qr. = 28 lbs.
1 hundredweight, cwt. = 4 qrs. = 112 lbs.
1 ton = 20 cwt. = 80 qrs. = 2240 lbs.

This is the system of weights in common use for the sale and preparation of pharmaceutical substances; the grain, ounce, and pound are recognised in the British Pharmacopœia. In addition to this the old apothecaries' weight is commonly used in prescriptions.

Apothecaries' Weight.

1 grain, gr.
1 scruple, ℈ = 20 grains.
1 drachm, ʒ = 3 scruples = 60 grains.
1 ounce, ℥ = 8 drachms = 24 scruples = 480 grains.
1 pound, ℔. = 12 ounces = 96 drachms = 288 scruples = 5760 grains.

The grain in each system is equal, consequently the ounce of avoirdupois is less than the ounce apothecaries' (or troy) by 42½ grains. It should also be noticed that the symbols for the ounce and pound differ in the two systems.

Imperial Measure of Capacity.

1 minim, ♏
1 fluid drachm, fl. drm. = 60 ♏.
1 fluid ounce, fl. oz. = 8 fl. drs. = 480 ♏.
1 pint, O = 20 fl. oz. = 160 fl. drs. = 9600 ♏.
1 gallon, C = 8 pints = 160 fl. oz.

The symbol O represents the *Octarius.*
,, C ,, *Congius,* a Roman measure equal to about six imperial pints.

The fluid ounce is the measure of an avoirdupois ounce of distilled water at 60° F., and consequently contains 437·5 *grains of water* at that temperature. It is very common to hear the grain and the minim confused; as a matter of fact the grain and minim are related as 1 to 0·91 approximately. The gallon contains 10 lbs. of distilled water at 60° F. = 70,000 grains, and consequently the pint equals 1¼ lbs.

At the present time the fluid ounce in use in the United States contains 453·7 grains, and the pint = 16 fl. oz., the gallon containing 8 pints; the same was the case in this country during the time that the old wine measure was employed, previous to the introduction of the imperial measure in 1824.

Measures of Length.

1 inch, in.
1 foot, ft. = 12 inches.
1 yard, yd. = 3 ft. = 36 inches.

The standard of length is that of a pendulum vibrating seconds of mean time in the latitude of London in a vacuum at the level of the sea = 39·1393 inches.

Relations of English Systems of Weight and Measure.

1 minim	is equivalent to		0·91 grain	of water	at 60° F.
1 fl. drm.	,,		54·68 grains	,,	,,
1 fl. oz.	,,	1 oz. or	437·5	,,	,,
1 pint	,,	1¼ lbs. or	8750	,,	,,
1 gallon	,,	10 lbs. or	70,000	,,	,,

1 cubic inch of water at 15·5° C. (60° F.) weighs 252·5 grains.

Relations of English and Metric Systems.

1 gramme	contains	approximately	15·432 grains.
1 litre	,,	,,	1·76 pints.
1 metre	,,	,,	39·37 inches.
1 are	,,	,,	119·6 square yards.

The student should work out a number of problems involving these relationships; for his guidance the following rules are given:

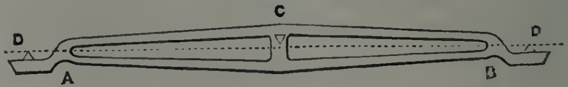
To convert	grammes	into	grains	multiply by	15·432.
,,	litres	,,	pints	,,	1·76.
,,	metres	,,	inches	,,	39·37.
,,	ares	,,	sq. yards	,,	119·6 as above.
,,	grains	,,	grammes	divide by 15·432, and similarly for litres, &c.	

To convert	c. centimetres (millilitres)	into minims	multiply by	16·93.
,,	kilogrammes	,, pounds	,,	2·2046.
,,	c. inches	,, c. centimetres	,,	16·37.

Scales and Balances.—Balances or scales are made on a multitude of patterns, and it is evident that the instrument intended for weighing small quantities of powerful poisons must be constructed very much more delicately than that used for large quantities of common substances.

It will be unnecessary here to do more than mention the various parts of a balance or scales, and the means of proving their accuracy or otherwise. The accompanying figure (Fig. 5) shows the essential parts. A B is the *beam*, supported by the *central knife-edge*, C, just above its centre of gravity upon a suitable support. D D are the *end knife-edges*, in this case exactly equidistant from C, and from which the *scale-pans* are suspended by wire or silk attached to plane surfaces resting upon D D.

Fig. 5.



A good balance will fulfil the following conditions:

1. The beam will be *light in weight* and *inflexible.* Lightness is necessary

for delicacy; inflexibility is important because any bending of the beam would lower the end knife-edges and tend to inaccuracy.

2. The centre of gravity should be slightly *below* the central knife-edge; this renders the balance sensible to very small weights.

3. The *end knife-edges* must be *exactly equidistant* from the *central knife-edge.* If this be unfulfilled the balance will be totally inaccurate—the longer arm will require a smaller weight than the shorter one to establish equilibrium. This, of course, only applies to equal-arm balances.

4. The three knife-edges must all be *parallel.* If this be unfulfilled the balance will not be *consistent;* that is, different weighings of the same substance will vary, according as it is placed in the centre or towards the edge of the scale-pan.

To test a balance—

1. The smallest weight to which the balance is required to turn is placed in one of the pans; it should immediately turn the balance.

2. Both pans being loaded with their full weight, the smallest weight is again placed in one of the pans; the equilibrium should be at once destroyed.

3. Equal weights being placed in each pan, they are then reversed, and should still be in equilibrium, showing equality of the two arms of the beam.

4. Equal weights being in each pan, one of the large weights is moved to various positions on the pan; no alteration should occur in the equilibrium, indicating that the knife-edges are parallel.

Balances for analytical purposes or for the finer weighing in dispensing are constructed most delicately, and should be carefully preserved from dust by enclosure in a glass case; a small quantity of lime is kept in the case to absorb damp or acid vapours; the pans should be carefully wiped with a soft chamois leather each morning before use. Weights should never be added to a delicate balance while in equilibrium; it should *be brought to rest* before adding or removing any weight.

Scales which are intended for large quantities are frequently constructed with one arm long and one short, the short arm being connected with a platform of iron or wood upon which the substance to be weighed is placed; a weight slides along the long arm, which is marked in a number of equal divisions; it is also terminated by an arrangement for receiving weights. The sliding weight when at the extreme end of the arm is equal to the smallest loose weight, and by bringing nearer to the other end its value is lessened; *e. g.* if it weighs 14 lbs. at the extremity it will be 7 lbs. in the middle, and so on. By this means large quantities can be weighed by the use of comparatively small weights, for if the short arm be only one tenth of the length of the long one, it will require ten times as much on the platform to produce equilibrium with a given weight on the extremity of the long arm.

Measures.—Measures for pharmaceutical use are constructed of glass, earthenware, copper, or tinned or enamelled iron. Small measures should be of glass, and are preferably of conical form, so that the smaller quantities may be measured in the narrowest part. A one- or two-drachm measure should be graduated at intervals of 5 to 10 minims, a two-ounce measure in fluid drachms, &c. For large measures metal is preferable on account of the danger of fracture of glass, but the want of transparency is a great drawback; they must be used with care to avoid alteration of volume by indentations. Earthenware or enamelled iron avoids this latter difficulty. A very convenient measure for quantities of 2, 4, or 8 pints may be made by measuring these amounts successively into a "pottle" bottle, and marking the level of the liquid *on the front and back* with a diamond. When measuring, care must be taken to have the surface of the liquid quite level, and the eye on a level with the surface. Most liquids adhere to the sides of the vessel, giving the appearance of two levels; this is called the *meniscus*, and the lower level is that to be observed.

Comminution of Drugs.—The comminution of drugs—that is, their reduction into smaller particles—is a matter of great importance; one of the main objects aimed at is to obtain a product of uniform fineness. If a drug is to be exhibited in the dry state it is usually required in very fine powder; if, however, it is to be made up into preparations such as tinctures, &c., it is necessary to reduce the drug to a more or less fine state of division, in order to facilitate the action of the liquids employed. The apparatus required varies according to the nature of the drug and the degree of division desired.

Comminution includes numerous operations, such as cutting, chopping, bruising, crushing, rolling, grinding, levigating, granulating, &c. The last of these will be treated under a special heading (*vide* "Precipitation"); the others will be most suitably described here.

Cutting, Slicing, and Chopping.—This process is employed when roots, &c., are required in a coarse state of division, its object being simply to cut across the drug so as to enable liquid solvents to penetrate the tissues; it is of special value in the case of such drugs as calumba, which contain much starch (*vide* "Infusion"). A cutter such as is shown in Fig. 6 is generally useful for operations on a small scale, those cutters which slice the root in a slanting direction being the most desirable.

On a larger scale, chaff-cutters, such as are used by farmers and others, may be employed for some soft herbaceous drugs.

Bruising or Contusion.—This operation consists in the reduction of a drug to powder, or simply cracking the drug, without powdering it, by a succession of blows. It is usually performed by means of a pestle and mortar, which, for the purpose of most organic drugs, may be made

FIG. 6.

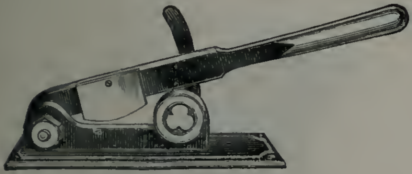
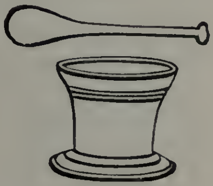


FIG. 7.



of bell-metal, brass, or cast iron. Fig. 7 represents the usual form of mortar employed; it must be sufficiently deep to prevent particles of drugs from being forcibly ejected, and, in the case of such drugs as ipecacuanha, which are specially liable to "jump," it is necessary to cover the mortar with a loosely fitting wooden lid, having a hole in the centre for working the pestle. The mortar should be set upon a firm foundation, such as a hard wooden block upon the ground-floor of the building, and, if of large size, must be secured by means of an iron band attached to and projecting beyond the top of the block, and fitting the bottom of the mortar. In order to reduce a drug to powder it is essential that it be thoroughly dried first, or the operation will be extremely tedious, and in many cases impossible. For moist drugs, or drugs containing much oil, such as almonds, fleshy roots in the fresh state, &c., a marble or wooden mortar is preferable; marble must not be used for drugs containing free acids. Mortars having pestles worked by machinery are usually called *stampers* (see Fig. 12, B, B).

Rolling and Crushing.—There is theoretically very little difference between these operations and that of *bruising*; in the latter case sharp percussion, and in the former sharp pressure attain the object aimed at, viz. a cracking, flattening, or partial powdering of the drug. These processes are usually carried out by means of special machines.

The essential part of a simple rolling-mill (Fig. 8) consists of two smooth granite, steel, or hard wooden rollers, similar to the rollers of a common mangle, but arranged side by side instead of one above the other. They can be made to revolve in the same or in opposite directions, and can be approximated near to each other or removed apart, according to the degree of crushing required. They are specially adapted for crushing fresh herbs or roots, and many other soft substances. When made with grooved or corrugated rollers they may be employed for grinding purposes.

FIG. 8.

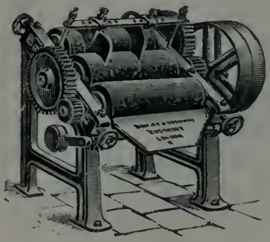
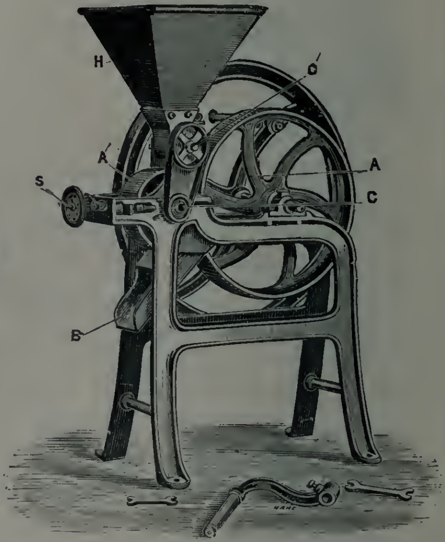


FIG. 9.



Mills for crushing oily substances, such as linseed, or for malt and many seeds, are usually made of a different pattern, but the same in principle. Fig. 9 represents a very useful form, which can be driven either by hand or steam power. The drug is delivered through the hopper H, between the wheels A and A′, which can be set at any desired distance apart by the screws. A and A′ revolve in opposite directions, and have flat but somewhat roughened edges. By the revolution of the wheels the seeds, &c., are drawn in between the two edges and crushed, but not powdered; two knife-edges scrape off any adherent drug from the wheels, and finally the crushed product is discharged through the shoot B. The wheel A′ is on the same axle as the small wheel C, which consequently turns the larger wheel C′, with which it is connected by a belt. This wheel C′ regulates the flow of the drug through the hopper.

Grinding.—Grinding has for its object the reduction of a drug to a uniform degree of comminution, which is attained by the agency of friction, or rubbing and crushing between level or cutting surfaces. What is sometimes termed powdering, pulverising, or trituration (in the special sense of rubbing to *powder*, not rubbing to *mix*) is really nothing more than grinding; it depends entirely on the same agency, the last being always conducted in a mortar. Grinding is the only means of obtaining a uniformly fine powder.

Many substances may be ground by simple rubbing with a pestle and mortar; these are usually the more friable chemicals, and such organic substances as some gums and resins; for these latter it is essential that they should be perfectly dry, and kept as cold as possible; asafœtida, for example, cannot be powdered in warm weather unless artificially cooled. The mortar employed for this purpose is composed of porcelain, Wedgwood-ware, composition earthenware, or glass. The operation is simply to rub the pestle in a circular or spiral manner upon the inner surface of the mortar, at the same time applying a firm downward pressure.

Drug-mills.—The great majority of drugs, however, are commonly ground by means of a mill. There are several kinds of hand drug-mills which are adapted for the use of the pharmacist; the most generally useful of these are the "Enterprise" mill and Hance's drug-mill. Both these possess the great advantage of being easily opened, so that the actual grinding surfaces can be cleaned. Fig. 10 shows the Enterprise mill, and Fig. 11 the same opened for cleaning. The following instructions for using the mill will explain its construction:—Fasten the mill with screws to any suitable fixture, with the drawer facing the operator. Now fit the axle of the mill into the grooves in the lower part of the body C, and draw over the upper half of the body, securing it by the thumb-screw B, which must be screwed down close. If properly arranged, and free from loose substances from a former grinding, the parts will now be arranged as follows :—The loose grinder D, which consists of a grooved inverted cone combined with a wide flange covered with "teeth," rests vertically by means of the projections

Fig. 10.

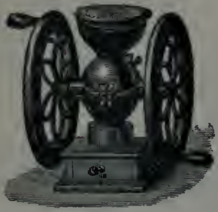
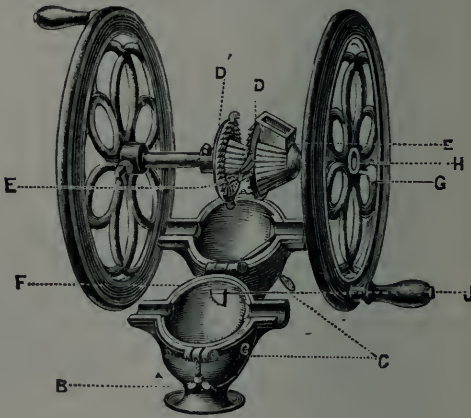


Fig. 11.



E, E, upon the bearers J, J. The grinder D′ is fixed to the axle, and fits into the loose grinder D, leaving space between the teeth for the passage of the drug when ground. The hopper (not shown in cut) delivers the drug into the oblong opening at the top of D.

The above instructions being carried out, the operator will now place a handful of material intended to be ground into the hopper, and give half a dozen turns. The ground drug will be delivered in the drawer. If too coarse or too fine, unscrew the thumb-screw G free from collar H, and turn the collar from or towards the operator, as the case may be; then screw the thumb-screw G into the hole in the side of the collar H.

Hance's drug-mill consists of a conical grinding surface set horizontally, and fitting into an inverted cone above. It is better adapted for heavy work than the Enterprise. It is driven by a fly-wheel connected with two bevelled cog-wheels, the second of which is on the shaft to which the conical grinding surface is attached. The fineness of powder is regulated by a thumb-screw, which elevates the revolving plate.

It must be remembered that for successful grinding the drug should be previously *dried* unless quite brittle; if damp, the mill will assuredly be clogged between the teeth, or at least the grinding will be greatly impeded. If a drug be in large pieces, it will save time and labour to first reduce it to a very coarse powder by setting the mill loosely, and then grinding again with the mill set at the required point.

By any of these means, however, it is practically impossible to reduce drugs to such a degree of fineness as is now commonly required for powdered drugs used for making the compound powders of the Pharmacopœia and similar purposes. This operation is now almost entirely relegated to the drug miller, who accomplishes it by means of chaser mills, roller mills, or buhr-stone mills, which will now be briefly described.

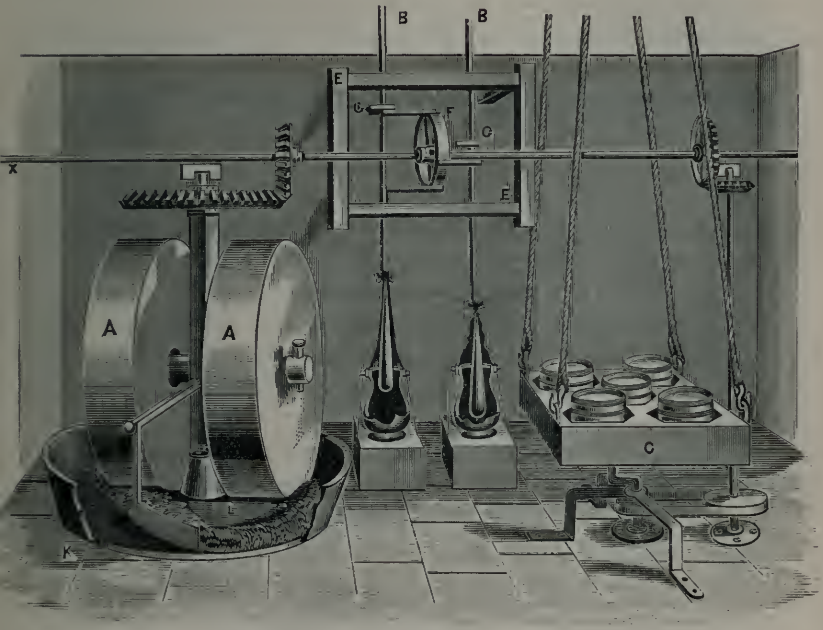


Fig. 12.—Drug-grinding apparatus.

A A. Chaser mill. B, B. Stampers. C. Sifting apparatus. E E. Guiding-frame for stampers. F. Revolving wheel, provided with projecting arms, G G, to raise stampers. The whole driven by the shaft X. K. The stone floor of mill room. L. Granite floor of mill.

Chaser mills consist of two heavy granite rollers (Fig. 12, A A), which are made to revolve upon their edges on a granite surface (L); they travel around the granite bed, which gives a grinding action on account of the outer portion of the surface of the roller having to travel through a larger circle than the inner, as well as the crushing effect of the weight of the stones. The drug, which has been pushed out of the path of the rollers, is continually shovelled back by a plough attached to the gearing, so that the whole is submitted to the grinding action. These mills are the most largely used for the production of very fine powders.

Roller mills have been already alluded to under "Crushing and Rolling;" for grinding purposes the rollers must be furrowed in an oblique direction.

Buhr-stone mills consist essentially of two stone discs, placed one upon another, which are secured so that the surfaces are almost in contact; one of the stones is stationary, the other revolves, and the substance is introduced between the two surfaces, where it is ground by the friction. The surfaces of the stones are crossed by furrows radiating to the circumference, so as to allow the powder to be passed outwards into a trough provided for its reception. The fineness of the powder is regulated by raising or lowering the moveable stones so as to bring them nearer or farther apart. They are mostly used for chemicals.

Disintegrators are frequently employed on the large scale; they are usually large mills, constructed somewhat on the principle of Hance's hand-mill; they will not, as a rule, grind very fine powders.

Sieves.—Whichever method of grinding is adopted, in order to obtain uniform powders it is necessary to sift the partially ground substance from time to time. Sieves are constructed of brass or iron wire, hair, or fine lawn; the wire or other gauze is stretched over a wooden frame, and secured in its place by a hoop of wood passing over the frame and gauze. Sieves are preferably enclosed with tightly fitting covers to both top and bottom, when they are called *drum sieves*. This precaution saves the annoyance and loss due to the flying away of fine particles of dust. Fig. 12, C, shows an arrangement for agitating a set of drum sieves. The degrees of disintegration of a powder are represented by the number of parallel wires of ordinary thickness within one linear inch forming the meshes of the sieve. Thus a No. 60 powder means a powder which will almost entirely pass through a sieve having sixty meshes to the linear inch, while scarcely any should pass through one having eighty meshes to the inch. The very fine powders of pharmacy are made to pass through a lawn sieve of 120 to 160 meshes to the linear inch. That which does not pass through the sieve is returned to the mill, and this alternate grinding and sifting is continued until nearly the whole of the drug has been powdered. Finally the sifted portions are lightly mixed in order to secure uniformity, the tough portion of a drug being the last to be divided into fine powder. There is almost always a small quantity of the tougher portions of a drug which remains in a coarse condition, and is called *gruffs*. This is usually reserved for a future operation, when it is added to the drug to be ground.

Mills are commonly cleaned by grinding several successive small quantities of sawdust, and then well brushing with a soft brush ; washing is not advisable if it can be avoided, as it damages the machinery, and renders the air of the mill room damp, but may be occasionally employed with advantage.

The following table gives the average loss sustained by drugs when partially dried and ground. It must be remembered that the loss varies enormously ; some bales of a drug will be quite soft and flexible, whereas others will break easily. The loss here shown does not include "gruffs," which are reserved for a succeeding operation ; it really shows only the loss by drying and by "dust." It must also be observed that the final powder is by no means perfectly dry, for if exposed to a temperature of 100° C. until it ceases to lose weight, a further loss of 5 to 10 or even 12 per cent. will be sustained. As examples of this further loss the following figures are given :—Cinchona, 7·9 per cent. ; gentian, 10·4 per cent. ; ipecacuanha, 7·3 per cent. ; jalap, 11·5 per cent. ; nux vomica, 10·0 per cent. ; opium, 7·5 per cent. ; stramonium leaf, 5·4 per cent.

Table showing Average Loss by Drying and Grinding.

Name of drug.	Loss per cent.	Name of drug.	Loss per cent.
Acacia	1·25	Coriandrum	2·4
Acidum Boricum	0·5	Cubeba	2·1
,, Citricum	0·2	Digitalis	4·4
,, Tartaricum	1·0	Fœniculum	2·4
Aconiti Radix	5·0	Galla	2·0
Aloë Barbadensis	2·8	Gentiana	7·0
,, Capensis	0·9	Glycyrrhiza	4·4
,, Socotrina	8·0	Guaiaci Resina	1·7
Ammoniacum	3·5	Ipecacuanha	3·0
Anisum	0·5	Iris Florentina	4·5
Areca	2·5	Jalapa	5·9
Asafœtida	3·0	Krameria	5·0
Aurantii Cortex	8·1	Lobelia	3·5
Belladonnæ Folia	6·6	Myristica	1·4
,, Radix	2·2	Myrrha	3·9
Borax	0·4	Nux Vomica	7·1
Calumba	2·7	Opium	13·5
Cambogia	1·6	Pimenta	1·1
Canella	2·2	Piper Nigrum	0·4
Cantharis	2·1	Potassii Tartras Acida	0·4
Capsicum	2·2	Rheum	2·5
Cardamomum	4·6	Scammonium	1·8
Cascarilla	5·0	Scilla	9·2
Catechu	1·6	Senega	3·5
Cinchona Flava	2·0	Senna	2·1
,, Pallida	2·7	Serpentaria	3·2
,, Rubra	1·5	Stramonii Folia	3·0
Cinnamomum	3·0	Tragacantha	3·5
Coccus	2·2	Valeriana	3·3
Colchici Semina	1·4	Zingiber	3·2

Levigation and Elutriation.—The terms levigation and elutriation are sometimes applied indiscriminately, but there is a material difference between them, although the principle is the same in each.

Levigation, or porphyrisation, is the operation of reducing a chemical or ore to an extremly fine powder by rubbing together with a liquid. It was commonly employed for grinding paints with oils, and similar purposes. The apparatus consists of a porphyry slab and a *muller,* which is similar to a short pestle with the lower end flattened. The substance under operation is placed on the slab in coarse powder, formed into a thick magma with the liquid. It is then distributed in a thick stratum, and triturated by means of the muller, which, being grasped with the two hands of the operator, is rubbed with some degree of pressure over the surface of the slab. In performing this operation the muller is made to describe in its course certain regular curvilinear figures, representing either a figure of eight or a series of intersecting circles; and these figures are alternated from time to time, so that by changing the direction in which the muller is moved a fresh set of particles may be brought under its action. In this way comminution is effected over an extended surface upon a very thin layer of the triturated substance, and the action is therefore more complete and uniform than that produced by the use of the pestle and mortar.

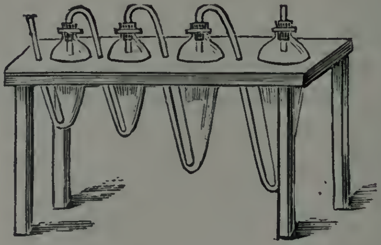
Elutriation consists in rubbing or grinding the chemical through the agency of a liquid, and when partly ground the coarser particles are allowed to settle for a few minutes, the supernatant liquid with finer particles poured off and allowed to stand for a long time until these finer particles have completely settled, when the liquid is again removed and the powder dried; the coarse particles are again ground with more liquid and treated as before, this operation of grinding and settling being repeated until the whole or nearly the whole of the substance is reduced to fine powder. Elutriation, therefore, entails washing, levigation does not necessarily do so. In some cases—as, for instance, prepared chalk and calamine—the whole of the mass is not reduced to fine powder, because the native mineral contains silica, which separates with the coarser particles, and can in this way be removed.

The following substances are usually pulverised by elutriation:—Calamina, B. P., and Creta Præparata, B. P.; also powdered charcoal, fuller's-earth, and pumice.

Fig. 13 shows a simple apparatus for elutriation. By keeping a stream of water flowing through the apparatus from left to right the finer particles are continually drawn over, the gritty and heavier portions being retained in the glass receivers.

Trochiscation.—Sometimes elutriated powders are dried in the form of little lumps of conical form; this operation is called *trochiscation.* The moist mixture of elutriated powder and water is transferred to an inverted metal cone having the apex open. This cone is supported on a movable frame, so arranged that by a succession of sharp taps small portions of the mixture are caused to fall through the cone on to a slab of some porous material, such as chalk, which rapidly absorbs their moisture and effects speedy drying. The conical lumps of prepared chalk, as sold in the shops, are produced in this way.

Fig. 13



Questions on Chapter III.

1. What parts of the following plants are used in pharmacy?—Aconite, belladonna, hemlock, colchicum, hops, mezereon, stramonium.

2: Draw up a list of precautions to be observed in drying fresh plants for use in medicine.

3. Explain why it is necessary to secure a good draught in the drying closet.

4. Express 55° C. in F. and in R. degrees. Why should thermometers be kept some time before use?

5. Give the equivalent in grains of ·5 gramme, 1·3 kilogrammes, and 3·5 milligrammes.

6. What is the relation between the metric measures of capacity and weight?

7. How many grains by weight are there in 13 fl. oz., 2 fl. drs., and 50 minims of distilled water at 60° F.?

8. How many gallons of water at 60° F. will weigh 1 ton 3 cwt. 0 qrs. and 4 lbs.?

9. How many cubic inches are there in 10 pints?

10. Express in grammes (*a*) 145 grains and (*b*) 1 lb. 3 oz., and in metric measure (*a*) 1 pint $12\frac{1}{2}$ fl. oz., (*b*) 9 fl. drs. 28 minims.

11. How would you determine the accuracy of a pair of dispensing scales (not the weights)?

12. What is meant by the following?—Comminution, contusion, grinding, levigation, elutriation.

13. Distinguish (*a*) the roller mill, (*b*) the chaser mill, (*c*) buhr-stone mill.

14. What is meant by a No. 40 sieve? How many holes will there be in 2 square inches of a No. 80 sieve?

CHAPTER IV

TRITURATION OF SOLIDS

Trituration is the act of powdering or mixing by rubbing together: the operation is usually performed in a mortar. It may be advantageously carried out as follows:—The pestle should be put into the middle of the mortar, the powders added according to directions; then revolve the pestle in gradually widening circles until the side of the mortar is reached, at the same time exerting a downward pressure. With a little practice the pestle can also be made to revolve on its axis in the hand, which materially assists the mixing operation. When using the right hand, most beginners triturate in the direction in which the fingers of a clock turn. It will be found, however, that the opposite direction is far preferable when once the habit is formed, as it is less tiring, and more power can be exerted. With the left hand, of course, the motion should be in the reverse direction.

We have previously noticed the use of trituration for the object of reducing substances to powder (p. 37); we are now concerned mainly with trituration for the purpose of mixing solids: it is also employed for mixing solids with liquids, or in some cases liquids only.

Compound Powders.—Small quantities of dry powders can readily be mixed by simple trituration, as described above. If, however, larger quantities than one pound are required, the object may usually be more rapidly accomplished by lightly mixing the powders, passing the mixture through a somewhat coarse sieve (Nos. 16 to 20), and finally lightly triturating. This last trituration is necessary because the larger particles of a powder will be the last to pass through the sieve, and would therefore render the sifted product deficient in uniformity.

The general rule for mixing powders by trituration is to mix those which are in smallest quantity first, then add the larger quantities gradually. Even when sifting is used to mix a compound powder, potent drugs such as opium must always be first thoroughly mixed with a portion of one of the other ingredients by trituration.

The compound powders of the British Pharmacopœia are sixteen in number; three or more of these should be prepared by the student or apprentice, commencing with a simple example, such as—

Pulvis Ipecacuanhæ Compositus.

Take of—

Ipecacuanha, in powder	1 part.*
Opium, in powder	1 ,,
Sulphate of potassium, in powder . . .	8 parts.

= 1 each of opium and ipecacuanha in 10.

Triturate the opium and ipecacuanha till thoroughly mixed, then add about one third of the sulphate of potassium, mix, add the remainder of the sulphate of potassium and mix thoroughly, then pass the whole through a fine sieve, and finally mix lightly in a mortar.

The following official compound powders may be mixed in the same way as compound powder of ipecacuanha :

Name of powder.	Proportions of ingredients.	Proportion of chief ingredient.	Notes.
Pulvis Scammonii Compositus	Scammony resin 4, jalap 3, ginger 1	1 in 2	1
Pulvis Catechu Compositus	Catechu 4, kino and rhatany, of each 2; cinnamon and nutmeg, of each 1	1 in 2½	2
Pulvis Antimonialis	Oxide of antimony 1, phosphate of calcium 2	1 in 3	—
Pulvis Cinnamomi Compositus	Cinnamon, cardamoms, ginger, of each 1	1 in 3	—

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* The word "parts" in such formulæ refers to parts *by weight* (ounces, pounds, grammes, &c.) unless stated to be *fluid parts*.

Name of powder.	Proportions of ingredients.	Proportion of chief ingredient.	Notes.
Pulvis Jalapæ Compositus	Jalap 5, acid tartrate of potassium 9, ginger 1	1 in 3	1
Pulvis Rhei Compositus	Rhubarb root 2, light magnesia 6, ginger 1	1 in 4½	1 and 3
Pulvis Cretæ Aromaticus	Prepared chalk 11, cardamoms 1, cloves 1½, saffron 3, nutmeg 3, cinnamon 4, sugar 25	1 in 4½ nearly	4
Pulvis Tragacanthæ Compositus	Tragacanth, acacia, and starch, of each 1; sugar 3	1 in 6	—
Pulvis Glycyrrhizæ Compositus	Senna and liquorice, of each 2; fennel fruit and sublimed sulphur, of each 1; refined sugar 6	1 in 6	2
Pulvis Opii Compositus	Opium 3, black pepper 4, ginger 10, caraway fruit 12, tragacanth 1	1 in 10	2 and 5
Pulvis Kino Compositus	Kino 15, opium 1, cinnamon 4	opium, 1 in 20	—
Pulvis Cretæ Aromaticus cum Opio	Opium 1, aromatic powder of chalk 39	1 in 40	—
Pulvis Elaterini Compositus	Elaterin 1, sugar of milk 39	1 in 40	6

Note 1.—Ginger is added to these powders to counteract the griping effect of the purgatives.

Note 2.—The B. P. directions that these powders are to be passed through a *fine* sieve must not be too strictly interpreted, for it is impossible to reduce such drugs as fennel, nutmeg, and caraway to a *fine* powder without greatly impairing their value by removal of the aromatic principles during the excessive drying which is needed. A No. 40 sieve is quite fine enough, and even this will probably reject a small proportion of these constituents.

Note 3.—The B. P. directs this powder, in common with most others, to be kept in well-closed bottles. This precaution is especially necessary in this case. as the powder is liable to absorb moisture (and carbonic acid) from the air, when combination takes place between the magnesia and one of the constituents of the rhubarb, with production of a pinkish colour. For the same reason this powder should not be mixed on a *damp day* if it can possibly be avoided. It is also well to store the powder in a dark cupboard, as strong light has the same effect as damp upon its colour.

Note 4.—Prepared according to the above directions, this powder will not possess the brilliant orange colour of the product of many manufacturers. This is attained by damping the saffron with spirit or water, and triturating with considerable pressure along with the chalk, then drying the mixture and adding the other ingredients.

Note 5.—The tragacanth is added to this powder on account of its use in preparing the confection of opium. It stiffens the preparation.

Note 6.—Elaterin being a most powerful drug, sugar of milk is added simply as a diluent, to enable exact doses to be more easily accomplished.

The two remaining compound powders of the Pharmacopœia (which must be prepared by the student) require special treatment as follows :

“*Pulvis Amygdalæ Compositus.*

“Take of—

Sweet almonds	8 parts.
Refined sugar, in powder	4 ,,
Gum acacia, in powder	1 part.

“Steep the almonds in water until their skins can easily be removed, and when blanched, dry them thoroughly with a soft cloth ; then rub them lightly in a mortar to a smooth consistence. Mix the gum and the sugar, and adding them to the almond pulp gradually, rub the whole to a coarse powder. Keep it in a lightly covered jar.”—B. P.

Either boiling or cold water may be employed to soften the skins, the former acting very much more quickly, from fifteen to thirty minutes being ample. After blanching and rubbing with a soft cloth it will be advantageous to put the almonds in a warm dry place for some hours (say overnight); by this means a drier powder will finally result. A marble or large composition mortar is most suitable for reducing the almonds to a paste ; they must be rubbed firmly but not *beaten*, or the oil will be partially separated, and will be less easily incorporated into a powder. The whole may be passed through a No. 10 or 12 sieve. If kept in a tightly closed bottle or jar it will become rancid.

“*Pulvis Sodæ Tartaratæ Effervescens.*

“Take of—

Tartarated soda, dried	120 grains.
Bicarbonate of sodium, dried	40 ,,

Mix, and wrap in blue paper.

Tartaric acid, dried	38 ,,

Wrap in white paper.”—B. P.

When taken, the contents of the blue paper are stirred up in about half a pint of water, the acid powder added, and the whole drunk while effervescing. The effervescence is due to the escape of carbonic acid gas. In drying the tartarated soda care must be taken not to over-heat it, or it will liquefy. If not dried, the papers will be stained by the powders.

There are some other official preparations made by trituration.

Hydrargyrum cum Cretâ.—(Grey powder.) Mercury, by weight, 1; prepared chalk, 2; = 1 in 3. The object is to obtain the mercury in such a fine state of division that globules are no longer visible to the naked eye, even if the powder be pressed out on paper. This is accomplished by firm trituration, or by prolonged agitation in a bottle.

Many of the ointments may be prepared by simple trituration in a mortar, or rubbing on a slab by means of a spatula. The consideration of this class of preparations will, however, be deferred to a later chapter, when the series may be conveniently studied together (see "Fusion").

Unofficial Powders.—In the preparation of tooth powders and similar articles, which are made in the same manner as B. P. compound powders, the ingredients must be in the finest powder possible; none should be used which will not completely pass through a fine lawn sieve of at least 120 meshes to the linear inch. If carmine or other colouring matter, or perfumes such as otto of rose, enter into the composition, these should first be well triturated with a small quantity of chalk or other powder, *and no further addition made until these are perfectly homogeneous*. This may be ascertained in the case of colouring matters by taking out a little of the powder on white paper, and pressing the flat surface of a spatula firmly on the powder, at the same time drawing the spatula along, so as to spread out the powder in a thin layer. If not mixed, the carmine will show streaks of darker tint than the other powder. If no streaks be seen, the other powders may be added (gradually at first), and the whole finally sifted. Camphorated chalk is a common dentifrice. The camphor may be reduced to powder if moistened with a very little spirit. Sugar also assists the powdering of camphor.

Questions on Chapter IV.

1. Define trituration. For what purposes is it employed?
2. Describe the method of preparation of compound powder of opium and aromatic powder of chalk; what is the object of the tragacanth in the former?
3. In what way and from what causes does compound rhubarb powder deteriorate?

4. Describe the preparation of compound almond powder. Why should it not be kept in tightly closed vessels?

5. What is the proportion of active ingredient in each of the following powders?—Pulv. Opii Co., Pulv. Elaterini Co., Pulv. Kino Co., Pulv. Ipecacuanhæ Co., Pulv. Jalapæ Co., and Hydrargyrum cum Cretâ.

CHAPTER V

SOLUTION

Many solid substances, when immersed in water or other liquids, more or less rapidly lose their solid form, and by simple agitation become evenly distributed throughout the whole body of liquid, so that the whole appears as one homogeneous fluid. The highest microscopic power is quite incapable of revealing the presence of the solid particles in such a mixture. Many familiar examples will occur to the student, such as the disappearance of sugar in a cup of tea. In such cases the water or other liquid is called the *solvent*, the substance dissolved is said to be *soluble*, and the liquid produced is the *solution*. The act of dissolving is also called *solution*. Substances incapable of thus dissolving in a given solvent are said to be *insoluble*.

Liquids and gases as well as solids possess this same property of solubility or insolubility; for example, alcohol and ammonia gas are very soluble in water, whereas mercury and oil are insoluble and nitrogen gas nearly insoluble in water.

Many liquids possess the power of dissolving one another in all proportions, *e. g.* alcohol and water, ether and chloroform; but in most cases they possess a definite degree of solubility, like the great majority of solids. When a solvent contains in solution the greatest possible proportion of a soluble substance it is said to be a *saturated* solution.

It is probable that no substance is *absolutely* insoluble in water, although a great many are so nearly so that for all practical purposes they may be regarded as insoluble, *e. g.* sulphate of barium, chalk, &c. All degrees of solubility are known between these extremes. Each substance has its own specific degree of solubility, which, however, varies with the temperature; for solids and liquids the solubility usually increases with the temperature; exceptions are, however, numerous, *e. g.* hydrate of calcium (slaked lime) is far more soluble in water at 15·5° C. than at 100° C. All gases are, however, more soluble in cold than in warm water.

Other circumstances besides temperature modify the amount of a substance dissolved by the solvent, *e.g.* the position of the substance in the solvent, the time allowed, and the fineness of division of the substance. If a solid body be introduced into a liquid in which it is soluble and allowed to remain undisturbed, it usually sinks to the bottom and there forms a strong solution; but this being heavier than the superincumbent solvent, little or none of the dissolved substance rises to the upper part. This is familiarly noticed in the case of a cup of tea, the sugar not being tasted unless stirred up. This fact renders it obvious that solution will take place more rapidly if agitated, or if the solid be suspended in the upper part of the liquid, so that the heavy solution can fall to the bottom and be replaced by fresh solvent. If the solid be lighter than the solvent, it must be kept at the bottom by attaching it to a glass rod or other suitable body.

Time, too, has much influence; as a rule the longer the contact the stronger the solution becomes until the point of saturation is reached.

The finer the powder the more rapidly is solution effected. It is evident that a vast number of small particles will present an immensely greater surface to the action of a solvent than a single large piece of the same weight; hence the rapid solution of the fine powder.

Although solution may be very reasonably considered to be something more intimate than mere mixture, such cases as those cited above may be called *simple* solution, as distinct from those in which a manifest chemical change takes place, as, for instance, when zinc is dissolved by diluted sulphuric acid. In the former class it is quite easy to obtain the original constituents without having recourse to anything beyond such simple means as evaporating the solvent; whereas in the latter zinc can never be recovered from the solution without submitting it to chemical treatment, in order to decompose the sulphate of zinc formed.

From these considerations the solutions of the Pharmacopœia may be conveniently classified as follows:

Group 1.—Solutions of liquids in another liquid.

Group 2.—Simple solutions of solids in a liquid.

Group 3.—Solutions of solids in a liquid involving chemical changes.

Group 4.—Solutions of gases in a liquid.

Group 1.—*Solutions of Liquids.*

To the casual observer the solution of one liquid in another is nothing more than simple mixture, but evidence to the contrary is abundant—such as contraction of volume and increase of temperature. A great many of the preparations of the Pharmacopœia are simple solutions of this character.

" *Spiritus Tenuior* (*Proof Spirit*).

Take of—

Rectified spirit	5 pints.	
Distilled water	3 ,,	Mix."

The name proof spirit is derived from the fact that it was formerly customary to test spirit by moistening gunpowder with it and setting fire to the spirit. It was found that spirit below this strength caused the combustion of the powder, and was said not to stand the "proof."

Rectified spirit is what is commonly termed spirit of 56° over proof (56° O. P.); that is to say, 100 volumes of rectified spirit require diluting to 156 volumes with water in order to produce proof spirit. When mixed with water, contraction of volume takes place; 60 volumes of water are therefore required to produce 156 volumes of proof spirit.

Spirit of other strengths are common articles of trade, especially 65° O. P., which corresponds with the alcohol of the United States Pharmacopœia. Rectified spirit contains 84 per cent., proof spirit 49 per cent., and 65° O. P. spirit 94 per cent. of absolute alcohol. The British Pharmacopœia requires that proof spirit shall have a specific gravity of ·920.

Specific Gravity.—Common observation will have taught the student that a given volume of one substance weighs considerably more than the same volume of another. Unless otherwise stated, it is customary to indicate the relative weights of all solids and liquids in comparison with distilled water at 15·5° C. as 1·000. This relative weight of any substance is called its *specific gravity. The specific gravity of any substance is, therefore, the weight of a given volume at* 15·5° *C. in comparison with the weight of the same volume of distilled water at* 15·5° *C.* It is important to observe that the bulk of almost every known substance is increased by an increase of temperature, consequently the specific gravity is less, the higher the temperature.

The determination of specific gravity is an operation of the greatest importance; it is frequently necessary to know the specific gravity during the manufacture of pharmaceutical preparations, and also as an indication of the purity or strength of the finished products. The principal methods of ascertaining the specific gravity of liquids will therefore be described.

Three principal methods are commonly employed—

1. *By the sp. gr. bottle.*—A small flask (preferably stoppered) is selected, having a very narrow neck, and capable of holding from 25 to 100 c.c., or 250 to 1000 grains. It is first thoroughly washed, rinsed with distilled water, and dried: the drying is best effected by heating over a small smokeless flame (or in air oven), and aspirating the steam by means of a glass tube. It is accurately weighed on a delicate balance, and the weight recorded. Now fill it about halfway up the neck with distilled water at exactly 15·5° C., and weigh again. By subtracting the weight of the empty flask from that of the flask and water, the weight of the water only is obtained. The simplest way to ensure the correct temperature is to insert the flask and contents in a basin of water at 15·5° C. a few minutes. For convenience it is advisable to adjust the quantity to an exact number of grammes or grains by removing or adding water. The sides of the neck of the flask must be carefully dried by means of a coil of blotting-paper, which also serves to remove any superfluous liquid. When a suitable weight of water has been obtained, the neck of the flask is scratched by a diamond at the exact level of the lower edge of the curved surface of the water. The flask is then emptied and rinsed twice with the liquid, the sp. gr. of which is required,

if miscible with water, or dried if immiscible; now fill the flask to the mark with the liquid, taking care that the temperature is correct, and dry the surface of the neck, then weigh again. The weight of liquid divided by that of the water gives the specific gravity. An example will make this clearer.

	Grms.		Grms.
Wt. of flask and water	39·834	Wt. of flask and proof spirit	37·834
Wt. of flask . .	14·834	Wt. of flask . . .	14·834
Wt. of water	25·000	Wt. of proof spirit	23·000

25) 23·000 (·920 = sp. gr. of proof spirit.

225

50

50

Sp. gr. bottles or flasks can be purchased, and are made in various forms; the two most useful are those figured. Fig. 14 is Renoualt's, and is used as described above. That shown in Fig. 15 is provided with a perforated stopper; it is completely filled, and the stopper inserted, when the excess of liquid rises through the perforation, and is removed from the surface of the stopper before weighing. Sp. gr. bottles when bought ready made should always be carefully checked before use, as they commonly vary to a small extent from the recorded weights. They should be further checked from time to time, as the volume will vary slightly during the first few months, after which it becomes constant.

2. *By the Sprengel tube.*—A "Sprengel tube" may be used when the quantity of liquid is limited. It consists of an elongated **U**-tube, the open ends of which terminate in two capillary tubes, A, B, which are bent at right angles in opposite directions (Fig. 16). It is filled by suction, an india-rubber tube being applied to the end B, and the liquid drawn up to the mark C. It is used just as the bottle, the temperature being adjusted by immersion in water at 15·5° C.*

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* Vide 'Pharm. Journ.' (3), iv, p. 85. Sprengel's original tube was *completely* filled, but the author prefers to fill it to a certain mark only, thus avoiding inconvenience often experienced in warm weather owing to the expansion of the liquid.

3. *By the hydrometer.*—This method is very expeditious, but not sufficiently accurate for many purposes, although very valuable for such liquids as syrups, which do not require great accuracy.

Fig. 14.

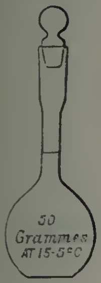


Fig. 15.

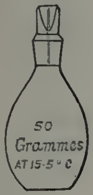


Fig. 16.

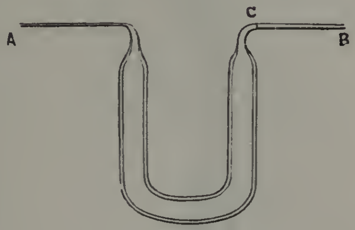
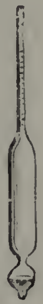


Fig. 17.



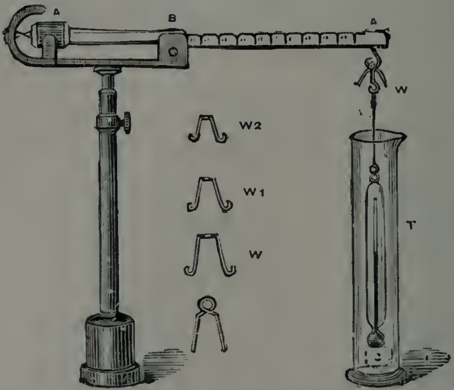
The hydrometer commonly in use (Fig. 17) consists of a floating vessel of glass, the lower portion of which carries a small bulb weighted with mercury, and the upper is elongated into a stem, which is divided into a scale indicating the sp. gr.

A tall glass cylinder is nearly filled with the liquid, and the hydrometer gently floated upon it, keeping it from actual contact with the side of the cylinder. The instrument will sink until it has displaced a volume of liquid of exactly its own weight, consequently the lighter the liquid the deeper will the hydrometer sink. As soon as it becomes stationary the number on the scale at the *surface* of the liquid is observed, and is the sp. gr. required. Hydrometers having a wide range should be rejected, as they are usually inaccurate, and instruments are readily procurable having a range not exceeding 200, *i. e.* from 1·000 to 1·200, or 1·200 to 1·400. Some hydrometers are graduated upon an arbitrary scale; such is Twaddell's, for the equivalents of which see Appendix.

4. *By the hydrostatic balance.*—This is an admirable instrument, and is now made by most makers of chemical balances. It furnishes by far the best general method of taking the sp. gr. of pharmaceutical preparations, as it is very expeditious and almost as accurate as the bottle.

It is shown in Fig. 18, the beam A A carrying the thermometer T, suspended by a fine platinum wire, balances horizontally upon the knife-edge at B. The liquid is placed in the cylindrical glass C; the thermometer being then immersed, the equilibrium is disturbed, and it will be necessary to place weights upon the beam in order to bring it again into the horizontal position. The weights are so adjusted that they represent directly the sp. gr. of the liquid. The large weight W, if hung upon the hook at extremity of beam, indicates 1·000, or if upon the grooves along the beam ·1, ·2, &c. The medium weight W1 represents ·01, ·02, &c.; and the small one ·001, ·002, &c. The thermometer serves to register the temperature as well as act as a plummet. The weights required will clearly be in proportion to the sp. gr. of the liquid, since the loss of weight sustained by the plummet will be the weight of a volume of the liquid equal to the volume of the plummet.

FIG. 18.



A rough method of finding the sp. gr. of syrups and other liquids is to accurately weigh an ordinary 10-oz. measure; carefully measure 10 fl. oz. of the liquid, and reweigh; the increase in weight divided by 10 gives the sp. gr.

The determination of the sp. gr. of solids and gases is not required as an operation of pharmacy, but is of service in the examination of many substances as a test of purity. The methods are described in most works on chemistry.

B. P. Preparations.—Other official preparations which are prepared by the simple mixture of two or more liquids are—

Name of preparation.	Formula in fluid parts.	Sp. gr.	Remarks.
Spiritus Ætheris	Ether 1, rectified spirit 2	·809	—
Spiritus Chloroformi	Chloroform 1, rectified spirit 19	·871	*Syn.* chloric ether.
Spiritus Cajuputi	Oil of cajuput 1, rectified spirit 49	·840	—
Spiritus Cinnamomi	Oil of cinnamon 1, rectified spirit 49	·842	—
Spiritus Juniperi	Oil of juniper 1, rectified spirit 49	·839	See note 1.
Spiritus Lavandulæ	Oil of lavender 1, rectified spirit 49	·839	—
Spiritus Menthæ Piperitæ	Oil of peppermint 1, rectified spirit 49	·839	—
Spiritus Myristicæ	Oil of nutmeg 1, rectified spirit 49	·839	—
Spiritus Rosmarini	Oil of rosemary 1, rectified spirit 49	·839	—
Essentia Anisi	Oil of anise 1, rectified spirit 4	·867 (about)	—
Essentia Menthæ Piperitæ	Oil of peppermint 1, rectified spirit 4	·8565 (about)	—
Enema Opii	Tincture of opium 1, mucilage of starch 32	—	—
Linimentum Chloroformi	Chloroform 1, liniment of camphor 1	1·215 (about)	—
Linimentum Opii	Tincture of opium 1, liniment of soap 1	·920 (about)	See note 2.
Linimentum Crotonis	Croton oil 2, oil of cajuput 7, rectified spirit 7	·899 (about)	See note 3.
Linimentum Terebinthinæ Aceticum	Glacial acetic acid 1, oil of turpentine 4, liniment of camphor 4	·914 (about)	See note 4.
Acidum Aceticum Dilutum	Acetic acid 1, distilled water 7	1·006	4·27 per cent. real acetic acid.
Acidum Nitro-hydrochloricum Dilutum	Nitric acid 3, hydrochloric acid 4, distilled water 25	1·07	See note 5.
Acidum Sulphuricum Aromaticum*†	Sulphuric acid 1½, strong tincture of ginger 1, spirit of cinnamon 1, rectified spirit 18	·926	See note 6. 13·8 per cent. sulphuric acid.
Aqua Chloroformi	Chloroform 1, distilled water 200	1·002	See note 7.
Liquor Ammoniæ	Strong solution of ammonia 1, distilled water 2	·959	—
Liquor Plumbi Subacetatis Dilutus*	Solution of subacetate of lead 1, rectified spirit 1, distilled water 78	—	See note 8.
Syrupus Aurantii	Tincture of orange 1, syrup 7	1·232 (about)	—

* To be prepared by the student.

† See 'Pharm. Journ.' (3), xx, p. 415.

Name of preparation	Formula in fluid parts.	Sp. gr.	Remarks.
Syrupus Zingiberis	Strong tincture of ginger 3, syrup 80	1·313	—
Tinctura Chloroformi Composita	Chloroform 1, rectified spirit 4, compound tincture of cardamoms 5	·964 (about)	—
Vapor Acidi Hydrocyanici	Hydrocyanic acid 1 to 1½, cold water 6	—	—
Vapor Coninæ	Hemlock juice 1, solution of potash ¼, distilled water 2	—	See note 9.
Vapor Creasoti	Creasote 1, boiling water 320	—	—
Vapor Iodi	Tincture of iodine 1, water 8	—	—
Collodium Flexile	Collodion 48 fluid parts, Canada balsam 2 parts, castor oil 1 part	800	—

Note 1.—If English oil be used, as directed in the Pharmacopœia, this preparation will be slightly milky in appearance.

Note 2.—This preparation throws down a deposit of meconate of sodium on keeping.

Note 3.—The cajuput oil used must not be of lower sp. gr. than ·920, or the resulting liniment will be cloudy, or even separate into two layers in cold weather.*

Note 4.—Acetic acid was formerly used in place of glacial acetic acid; the preparation was not then clear.

Note 5.—Must be kept fourteen days prior to use to ensure complete reaction between the acids. It contains when finished free chlorine, hydrochloric, nitric, and nitrous acids, &c.

Note 6.—The B. P. directions are—"Mix the sulphuric acid gradually with the spirit, and add the spirit of cinnamon and tincture of ginger." Great heat is produced, and unless the acid be added *very* gradually much loss will occur, and the colour of the preparation will be impaired. The B. P. erroneously gives sp. gr. ·911, and the percentage of acid as 12·5.

Note 7.—It requires violent agitation to effect solution.

Note 8.—The water must be boiled immediately before use to expel dissolved carbon dioxide, which would cause a cloudiness in the liquid by formation of carbonate of lead.

Note 9.—The potash is added to liberate free conine from the extract.

Many preparations made by simple admixture suffer such diminution of volume in the operation that the Pharmacopœia directs that the volume shall be adjusted when the mixing is completed and the liquid cooled to 15·5° C. They are the following:

* See 'Pharm. Journ.' (3), xx, p. 415.

Name.	Formula in fluid parts.	Sp. gr.	Strength.
Acidum Hydrochloricum dilutum	8 of strong acid, made up to 26½ with distilled water	1·052	10·58 per cent. real HCl.
Acidum Lacticum dilutum	3 of strong acid, made up to 20 with distilled water	1·040	12·9 per cent. real $HC_3H_5O_3$.
Acidum Nitricum dilutum	6 of strong acid, made up to 31 with distilled water	1·101	17·44 per cent. real HNO_3.
Acidum Phosphoricum dilutum	3 of strong acid, made up to 20 with distilled water	1·080	13·8 per cent. real H_3PO_4.
Acidum Sulphuricum dilutum	7 of strong acid, made up to 83½ with distilled water	1·094	13·65 per cent. real H_2SO_4.
Note 1.—Liquor Ammonii Acetatis	1 of stronger solution, made up to 5 with distilled water	1·017	7·45 per cent. real $NH_4C_2H_3O_2$.
Note 1.—Liquor Ammonii Citratis	1 of stronger solution, made up to 4 with distilled water	1·062	13·6 per cent. real $(NH_4)_3C_6H_5O_7$
Liquor Ferri Acetatis	1 of stronger solution, made up to 4 with distilled water	1·031	7·4 per cent. of $Fe3(C_2H_3O_2)$.
Liquor Ferri Perchloridi	1 of stronger solution, made up to 4 with distilled water	1·110	12·8 per cent. of Fe_2Cl_6 (about).
Tinctura Ferri Acetatis	1 of stronger solution, with 1 of rectified spirit, and ½ of glacial acetic acid, made up to 4 with distilled water	1·013	7·6 per cent. of $Fe3(C_2H_3O_2)$.
Tinctura Ferri Perchloridi	1 of strong solution, with 1 of rectified spirit, made up to 4 with distilled water	1·094	13·4 per cent. of Fe_2Cl_6 (about).
Liquor Trinitrinæ. Syn.—Liq. Nitroglycerini; Liq. Glonoini	Pure nitro-glycerine 1 part by weight, rectified spirit to make 100 fl. parts	·844	1 by weight in 100 by volume.

Note 1.—These must be stored in bottles free from lead, or they will become contaminated with that metal by dissolving it from the glass; the ordinary pale green glass bottles used by wholesale pharmacists are very suitable.

Most of these preparations become much heated in mixing, owing to chemical union between the strong acids, &c., with water; hence the necessity for allowing them to cool to 15·5° C. before finally adjusting the measurement. On this account also it is advisable always to *pour the strong acids into nearly the whole of the water at first* contained in a stoneware or *thin* glass vessel, not the water into the acid. The student should prepare diluted sulphuric acid, and at least one other of the above list; thin glass flasks, such as are sold by makers of chemical apparatus, are the most suitable vessels for mixing the dilute acids.

Group 2.—Simple Solution of Solids.

Solution of Solids.—The preparations made by this operation include a large number of the B. P. galenicals.

The student should prepare the following:

"*Liquor Calcii Chloridi.*

"Take of—

Chloride of calcium . . .	1 part.
Distilled water	5 fluid parts.

Dissolve and filter if necessary."—B. P.

Solution takes place quite readily in the cold. The act of solution is accompanied by a sensible rise of temperature. This is explained as follows:—Chloride of calcium exists in at least three forms: (1) anhydrous, having the formula $CaCl_2$; (2) the B. P. salt, which is dried at 400° F. (204·4° C.), and contains two molecules of water, formula $CaCl_2,2H_2O$; and (3) the ordinary crystalline salt, which contains six molecules of water, $CaCl_2,6H_2O$. When either the first or second of these compounds is dissolved in water, chemical combination occurs. In the case under notice four molecules of water are added to the $CaCl_2,2H_2O$ molecules, which chemical combination is sufficient to raise the temperature of the whole liquid. On the other hand, many substances, when dissolving, cause a diminution of temperature; this is due to the change from the solid to the liquid state, potassium nitrate and ammonium chloride are examples. See also "Latent Heat of Water."

Filtration.—Solution of chloride of calcium should be filtered if necessary. The object of filtration is to remove any undissolved particles of dust or other impurity, and allow the solution to pass through perfectly clear; this clear liquid is called the *filtrate*. In the case in point paper may be used, and no difficulty will be experienced; but in the whole range of pharmacy there is nothing which taxes the ingenuity and scientific skill of the pharmacist more than the *apparently* simple task of obtaining a perfectly bright filtrate. Instances illustrative of this will constantly appear during practical work. The act of filtration consists in bringing the more or less turbid liquid in contact with a porous substance, the fibres of which will retain the undissolved solid or oily matter, while the liquid passes through the pores. This is usually accomplished by putting the liquid upon the porous body contained in a funnel, when the clear liquid passes through by the force of gravity.

Funnels.—The accompanying figures represent different kinds of funnels: Fig. 20, a plain funnel; Fig. 21, a ribbed funnel; and Fig. 19 a badly constructed funnel. The angles at the points A, B, and C should all be 60°, or, if any variation be allowed, the angle at C should be rather less, say 58°, in which case those at A and B will be 61° each. Fig. 19 shows a funnel (common enough in the market) in which the angle at C is considerably greater than 60°. This is the worst possible form, for when the filter has been folded and filled with liquid the weight will press the lower portion tightly against the side of the funnel near the bottom, thus preventing the flow of liquid; whilst the upper portion of the filter is necessarily far away from the side, and having no support, it is liable to break longitudinally. The ribs in funnel, Fig. 21, are introduced with the object of keeping the paper from pressing too closely upon the funnel, and consequently they increase the rate of flow to some extent; but this is more readily accomplished by the use of one or more glass rods; they keep the paper well away from the sides, and, in most liquids, do not cause the filtrate to be less clear.

FIG. 19.

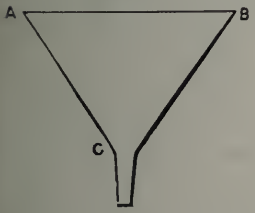


FIG. 21.

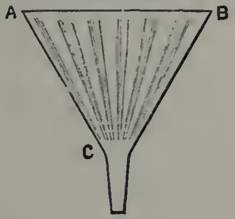
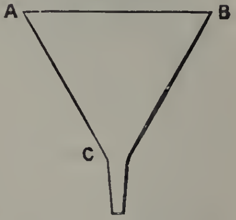


FIG. 20.



Funnels are usually made of earthenware or glass, although tinned iron, enamelled iron, copper, and gutta percha are also commonly employed. Metal funnels must be used with caution, as many liquids (acids, &c.) act chemically upon them.

Filtering Media.—The filtering media usually employed are paper, paper pulp, calico, linen, flannel, asbestos, glass-wool, and various inorganic substances, such as charcoal, fuller's-earth, talc, kaolin, pure silica or sand, or compounds of two or more of these. The paper used is unsized; it is sold as *filtering* paper, and can be bought ready cut into circular pieces of varying sizes, or in sheets. The best kind is Swedish, but this is used almost exclusively in quantitative analysis; for ordinary purposes white English (for colourless liquids) or grey French paper is employed. A special paper is made for use with liquids liable to burst the paper, having threads of linen at intervals of about one eighth of an inch woven into the papers. To use the paper it is folded either *plain* or *plaited.*

Plain filter.—A circular filter-paper is folded into a semicircle, then again into a quadrant; hold the folded paper point downwards, and open out so that one single thickness of paper is on one side, and three folds on the other. This is put into the funnel (with or without glass rods), and (usually) water poured on to thoroughly wet the paper; when drained, the liquid requiring filtration is introduced by carefully *pouring down the side of the filter where it is three-fold,* so as to avoid suddenly straining the apex of the filter, when the process of clearing will commence; in some cases it will be necessary to return the first portion of filtrate, as it may not be perfectly bright. When large filters are employed, or strongly acid or alkaline liquids, or those containing heavy solid matters in suspension, it will be well either to use a double filter, the second paper being so arranged that its three-fold half is adjacent to the single fold of the first paper; or at least to have a small filter-paper arranged as a cap to strengthen the apex of the cone. The folded filter-paper should be rather smaller than the funnel in which it is placed. Nothing is more slovenly than a projecting, badly cut, or torn filter; it wastes the liquid both by absorption and by evaporation, and worse still, it gets the operator into lazy and slovenly habits. The paper itself should not be *completely* filled with liquid; half an inch or so should be left unfilled, for without this precaution it will be found that many suspended solids will creep over the edge and cloud the filtrate.

The plaited filter.—The use of a plaited filter obviates the necessity for the use of glass rods to increase the rapidity of flow of the liquid.

This is made in the following manner:—A square piece of paper, *a b c d*, Fig. 22, is folded in the line *e f*, the edge *c d* being placed over *a b*. This doubled sheet is then creased as represented in the drawing. In the first place, the crease *g h* is produced by laying *b f* over *a e*, and pressing the thumb-nail, or any hard surface, over the folded edge so as to produce a sharp crease. Then placing *f* over *g*, the crease *b h* is formed; in like manner the crease *a h* is formed by laying *e* over *g*, and by similar means the intermediate creases *i, k, l, m*. These creases are all in one direction, forming seven receding angles, and in making them it is desirable not to bring the creases quite to the point *h*, but to leave about half an inch or less through which they do not pass; otherwise the frequent foldings of the paper at this point would so weaken the texture as to cause it to break with the weight of the liquid introduced into the filter. In the next place, an equal number of creases are to be made in the opposite direction,

FIG. 22.

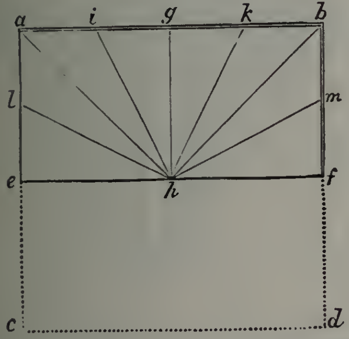
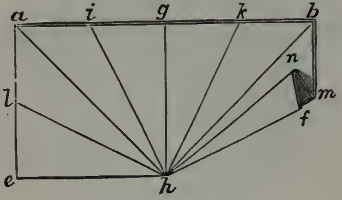


FIG. 23.



dividing each of the eight sections represented in the upper part of Fig. 22 in half. In doing this the edge *f h* is laid on the crease *b h* and then turned back as shown in Fig. 23, producing the crease *n h*. In like manner an intermediate crease is made in each of the other sections, so as to form a sort of fan, as represented in Fig. 24. The points *a*, *b*, are cut off with a sharp knife or scissors, and

Fig. 24.

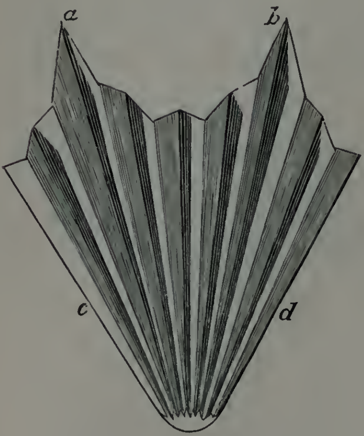
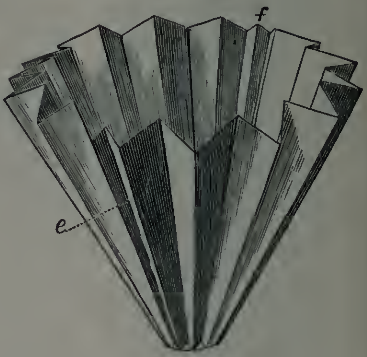


Fig. 25.



the filter opened to its proper angle by separating the originally doubled halves of the paper without disturbing the sharpness of the creases. It will now be found to consist of alternately projecting and receding angles, forming a uniform zigzag circumference, excepting at the points *c* and *d* (Fig. 24), at each of which places two projecting angles come together. The intermediate portion of paper between these two angles should be folded so as to form a small receding angle, as shown at *e* and at *f* (Fig. 25). This figure represents the appearance of the filter when completed.

When filtering into a bottle, care must be taken that there is a sufficient outlet for air between the neck of the funnel and that of the bottle, otherwise filtration will be retarded or even stopped. This object may be gained by having a small groove outside the funnel neck, or by inserting a piece of thick string between the funnel and bottle neck.

When filtering very volatile liquids, or liquids which require to be kept from exposure to the air, such as lime water, the funnel should be ground to a flat edge and covered with a ground glass plate, the air being permitted to pass from the receiving bottle into the funnel above the filter by using a glass tube in place of the glass rod as usual.

The use of other filtering media besides paper will be noticed when considering the preparations to which they may advantageously be applied, as well as special forms of apparatus, &c., *e.g.*:

For the use of paper pulp as a filtering medium, see Succi.

For the use of calico as a filtering medium, see Linimentum Camphoræ.

For the use of flannel as a filtering medium, see Syrupus.

For the use of asbestos as a filtering medium, see Liq. Acidi Chromici.

For the use of silica or pumice as a filtering medium, see Extractum Opii Liquidum and Glycerinum Aluminis.

For the use of animal charcoal as a filtering medium, see Alkaloids.

For hot filtration, see Glycerinum Aluminis and Adeps Præparatus.

For upward filtration, see Ext. Cascaræ Liquidum.

Name of preparation.	Formula.	Approximate sp. gr.	Remarks.
Glycerinum Acidi Carbolici	Carbolic acid 1 part, glycerine 4 fl. parts	1·219	Rub together.
Glycerinum Acidi Gallici	Gallic acid 1 part, glycerine 4 fl. parts	1·286	See note 1.
Glycerinum Acidi Tannici*	Tannic acid 1 part, glycerine 4 fl. parts	1·292	See note 1.
Glycerinum Aluminis	Alum, in powder, 1 part; glycerine 5 fl. parts	1·297	See note 2.
Glycerinum Amyli*	Starch 1 part, glycerine 5 fl. parts, water 3 fl. parts	—	See note 3.
Glycerinum Boracis	Borax, in powder, 1 part; glycerine 4 fl. parts, water 2 fl. parts	1·214	See note 4.
Injectio Apomorphinæ Hypodermica	Hydrochlorate of apomorphine 2 parts, camphor water 91 fl. parts	—	See note 5 = 1 grain in 50 minims.
Injectio Ergotini Hypodermica	Ergotin 1 part, camphor water 2 fl. parts	—	See note 5. = 1 in 2·7 fl. parts
Linimentum Camphoræ	Camphor 1 part, olive oil 4 fl. parts	·9275	See note 6.

Name of preparation.	Formula.	Approximate sp. gr.	Remarks.
Linimentum Camphoræ Compositum	Camphor 1 part, oil of lavender $\frac{1}{20}$ fl. part, strong solution of ammonia 2 fl. parts, rectified spirit 6 fl. parts	·869	—
Linimentum Iodi	Iodine 5 parts, potassium iodide 2 parts, glycerine 1 part, rectified spirit 40 fl. parts	·980	See notes 7 and 10.
Linimentum Sinapis Compositum	Oil of mustard 1·4 fl. parts, extract of mezereon 1 part, camphor 3 parts, castor oil 7 fl. parts, rectified spirit 44 fl. parts	·865	See note 7.
Liquor Acidi Chromici	Chromic acid 1 part, distilled water 3 fl. parts	1·185	See note 8.
Liquor Arsenicalis*	Arsenious acid 1 part, carbonate of potassium 1 part, compound tincture of lavender 3$\frac{1}{7}$ fl. parts, water to 100 fl. parts	1·010	See note 9.
Liquor Arsenici Hydrochloricus	Arsenious acid 1 part, hydrochloric acid 1$\frac{1}{4}$ fl. parts, distilled water to make 100 fl. parts	1·010	See note 9.
Liquor Atropinæ Sulphatis	Atropine sulphate 1 part, camphor water 99 fl. parts	1·003	See note 5.
Liquor Cocainæ Hydrochloratis	Cocaine hydrochlorate 1 part, salicylic acid ·015 part, distilled water to make 10 fl. parts	1·027	See note 5.
Liquor Calcii Chloridi	Chloride of calcium 1 part, distilled water 5 fl. parts	1·145	See above.
Liquor Hydrargyri Perchloridi	Perchloride of mercury 1 part, chloride of ammonium 1 part, distilled water 875 fl. parts	1·001	See note 11. = $\frac{1}{2}$ grain in 1 fl. oz.
Liquor Iodi	Iodine 1 part, iodide of potassium 1·5 parts, distilled water q. s. to produce 20 fl. parts	1·0955	See note 10.
Liquor Morphinæ Acetatis	Acetate of morphine 1 part, diluted acetic acid 2 fl. parts, s. v. rect. 25 fl. parts, distilled water q. s. to produce 100 fl. parts	·977	See note 11.
Liquor Morphinæ Hydrochloratis*	Hydrochlorate of morphine 1 part, diluted HCl 2 fl. parts, rectified spirit 25 fl. parts, distilled water q. s. to produce 100 fl. parts	·977	See note 11.
Liquor Morphinæ Sulphatis	Sulphate of morphine 1 part, rectified spirit 25 fl. parts, distilled water q. s. to produce 100 fl. parts	·977	—
Liquor Potassii Permanganatis	Permanganate of potassium 1 part, distilled water to produce 100 fl. parts	1·009	See note 11.
Mistura Sennæ Composita*	Sulphate of magnesium 8 parts, liquid extract of liquorice 2 fl. parts, tincture of senna 5 fl. parts, compound tincture of cardamoms 3 fl. parts, infusion of senna 30 fl. parts	1·095	= 1 in 5 fl. parts. Dissolve $MgSO_4$ in inf. sennæ; add extract and tinctures.
Mucilago Acaciæ*	Gum acacia 2 parts, distilled water 3 fl. parts	1·166	See note 12.

Name of preparation.	Formula.	Approximate sp. gr.	Remarks.
Mucilago Amyli*	Starch 1 part, distilled water 36½ fl. parts (nearly)	—	See note 13.
Mucilago Tragacanthæ	Tragacanth, in powder, 12 parts; rectified spirit 22 fl. parts, distilled water 875 fl. parts	—	See note 14.
Oleum Phosphoratum	Phosphorus 1 part, almond oil 99 parts	920	See note 15.
Spiritus Camphoræ	Camphor 1 part, rectified spirit 9 fl. parts	850	—
Syrupus*	Refined sugar 2 parts, distilled water 1 part	1·330	See note 16.
Syrupus Aurantii Florum	Orange-flower water 1 fl. part, sugar 6 parts, distilled water 2 fl. parts	1·330	See note 16.
Syrupus Chloral	Chloral hydrate 80, distilled water 82, syrup q. s. to 437½ fl. parts	1·320	= 10 grains in 1 fl. drm.
Syrupus Scillæ	Vinegar of squill 1 fl. part, sugar 2 parts	1·345	See note 16.
Tinctura Cannabis Indicæ	Extract of Indian hemp 1 part, rectified spirit 20 fl. parts	·866	Dissolve; filter if necessary.
Tinctura Iodi*	Iodine 1 part, potassium iodide 1 part, rectified spirit 40 fl. parts	·877	See note 10.
Tinctura Nucis Vomicæ	Extract of nux vomica 1 part, distilled water 13⅙ fl. parts, s. v. r., q. s. to produce 65¾ fl. parts	·890	Dissolve and filter.
Tinctura Podophylli	Podophyllin 1 part, rectified spirit 54·7 fl. parts	·845	= 1 grain in 1 fl. drm.
Tinctura Quininæ	Hydrochlorate of quinine 1 part, tincture of orange 54·7 fl. parts	·940	= 1 grain in 1 fl. drm. See note 17.
Tinctura Tolutana	Balsam of tolu 1 part, rectified spirit q. s. to produce 20 fl. parts	·880	—
Vapor Chlori	Chlorinated lime with cold water q. s. to moisten	—	Not to be filtered.
Vinum Antimoniale	Tartarated antimony 1 part, sherry 219 fl. parts	—	See note 18.
Vinum Ferri Citratis	Citrate of iron and ammonia 1 part, orange wine 54·7 fl. parts	—	= 1 gr. in 1 fl. drm. See note 19.
Vinum Quininæ*	Sulphate of quinine 1 part, citric acid 1½ parts, orange wine 437½ fl. parts	—	= 1 gr. in 1 fl. oz. See note 19.

This series of preparations illustrates the *great importance* of clearly distinguishing between the *minim* and the *fluid grain*. Those marked * to be prepared by the student.

Note 1.—The B. P. directs the heat of a water-bath for these preparations. Glycerine of tannic acid can be made without heat if longer time be allowed, and is then slightly paler in colour. Glycerine of gallic acid, if heated to

190° C., produces pyrogallic acid. They do not need filtration. Much of the glycerine of commerce contains iron ; this will cause discoloration of these preparations.

Water-bath.—The water-bath is described in the B. P. as "an apparatus by means of which water or its vapour, at a temperature not exceeding 212° F. (100° C.), is applied to the outer surface of a vessel containing the substance to be heated, which substance may thus be subjected to a heat near to, but necessarily below, that of 212° F." The water-bath is a most useful piece of apparatus, and is used in most operations requiring a low temperature, *i. e.* when the substance to be heated is liable to decompose or become discoloured by greater heat. Fig. 26 shows a convenient form. A is a copper trough which contains the water ; it is fitted with a tap B. It is convenient also to have a glass gauge to indicate the level of the liquid, and give timely warning of the need of a further supply of water. Water is added through the covered spout C. The top of the trough is fitted with copper rings, so that dishes of different sizes can be placed on the bath. The dish D fits into the bath or ring. The dishes may be of tinned copper, enamelled iron, porcelain, earthenware, glass, or, for analytical purposes, platinum, according to the nature of the substance to be heated. For glycerine of tannin, porcelain is the most suitable. Metal dishes should not be used for liquids containing mineral acids ; and if weak acids such as acetic be present, it is safest to use enamelled iron or porcelain, for if the tin be at all removed from the copper surface, solution of the copper will in many cases take place. Heat is applied by means of gas burners, a coke furnace, or steam, accord-

Fig. 26.

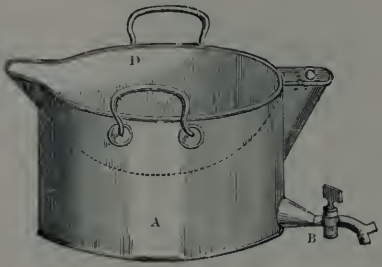
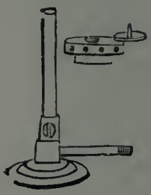


Fig. 27.



ing to the size of the water-bath. For small baths the ordinary Bunsen burner is suitable (Fig. 27); in this burner the gas passes upwards out of a narrow tube into a broad vertical metal pipe, in the lower part of which two or more large holes are made to admit air; these holes are just below the gas jet, and as the gas rises it draws air into the broad tube, the mixed air and gas being lighted at the upper end. The holes can be opened or closed and the air-supply regulated by means of a metal collar, so that the flame is either a plain gas flame or an air gas flame at will. The rose should be placed over the tube to distribute the heat when a large flame would be liable to fracture the vessel.

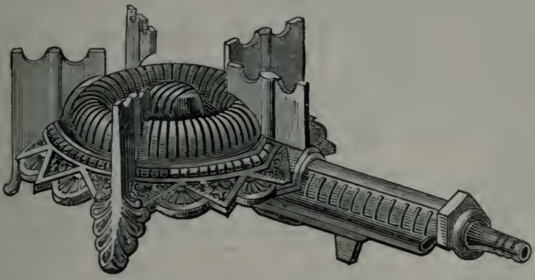


Fig. 28.—The "Radial."

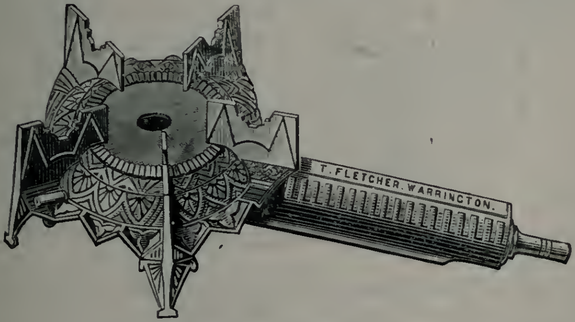

T. FLETCHER. WARRINGTON.

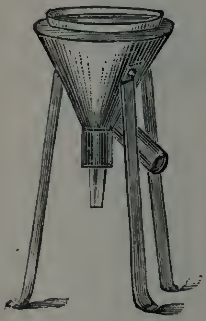
Fig. 29.—High power burner.

Two other useful gas burners, giving more heat than the Bunsen burner, are the "Radial" burner and Fletcher's high power burner; the form of these is shown in Figs. 28 and 29. The use of steam will be described in a future chapter.

Note 2.—Ammonia alum dissolves more rapidly than potash alum; this preparation is not easily filtered, it passes through paper cloudy. If, however, a very little pure silica be shaken up with the liquid before filtration it is obtained quite bright. The paper and silica must be free from iron, which is present in grey paper. It filters best while warm.

Hot Filtration.—Hot filtration is frequently a necessity or great advantage, as in the cases of glycerines, prepared lard, oxymels, and some syrups. For this purpose the funnel is placed in a hot-water jacket, which consists of a hollow metal funnel having a wide tube at one side; the tube and jacket are filled with water (or other liquid), and the whole kept hot by a flame applied to the end of the projecting tube of the jacket (Fig. 30). If a higher temperature than that obtainable in the above manner be required, it is best to support the funnel upon the ring of a retort stand, and heat it by means of a number of small gas flames arranged in a circular burner below the funnel.

FIG. 30.



Note 3.—The B. P. directions are—"Stir them together in a porcelain dish and apply heat, stirring constantly until the starch particles are completely broken and a translucent jelly is formed."

A convenient source of heat is the sand-bath.

Sand-bath.—The *sand-bath* consists of a thin iron or copper dish containing sand, on which the dish or flask is placed; it gives a more even distribution of temperature than an unguarded gas flame, consequently there is less risk of burning such substances as starch; it is used in cases in which a moderate heat is required. The three burners already mentioned may be used as the source of heat.

Note 4.—A partial chemical action occurs, boric acid being liberated with production of metaborate of sodium. Filter through paper while warm.

Note 5.—Camphor water or salicylic acid is employed as a preservative to prevent the growth of fungi. These may be filtered through a small plug of cotton-wool or glass-wool inserted in the neck of a funnel. The apomorphine hydrochlorate must be finely powdered to ensure solution. To prepare cocaine solution boil water, add acid, cool, dissolve cocaine, and make up to measure.

Note 6.—The solution of the camphor is accelerated if the oil be warmed;

this must be done in a loosely closed vessel, or some of the camphor will be lost, it being volatile. Strain through calico.

Filtration through Calico.—The calico strainer may be made either in the form of a conical bag having a *double* seam, and attached to a wooden ring supported by string from a beam or other suitable position, as shown in Fig. 31, or by simply stretching a calico square loosely over a frame standing upon four legs; it may be fastened by means of tacks or by string at the four corners (Fig. 32). The calico must be well rinsed with warm water to remove "dress," and dried before use for oils.

Fig. 31.

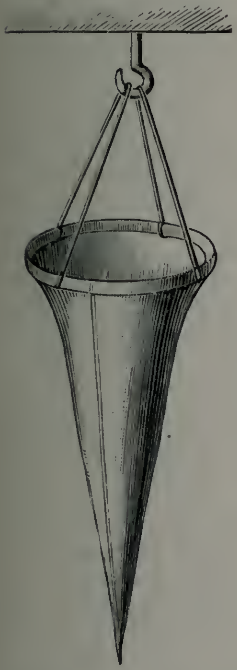


Fig. 32.

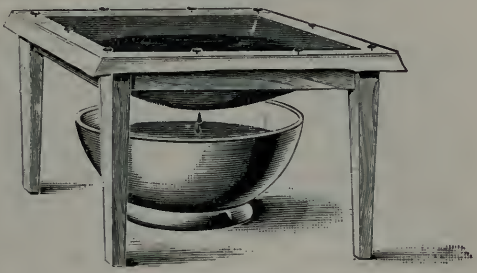
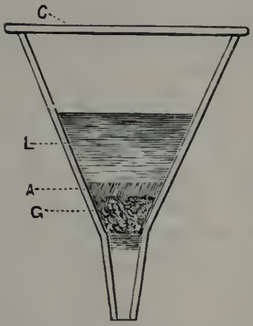


Fig. 33.



Note 7.—The glycerine or castor oil is added to prevent the liniments from drying too rapidly, and thus retaining the action of the iodine or oil of mustard during a longer period. The extract of mezereon assists the stimulant action of the oil of mustard in Lin. Sinapis Comp.

Note 8.—Chromic acid acts chemically upon organic filters, consequently asbestos or glass-wool should be employed.

Filtration through Asbestos.—Glass-wool may be simply used to plug the throat of the funnel, but asbestos requires more careful treatment. The lower portion of the funnel is first filled with coarsely broken glass or clean gravel (Fig. 33, G); sufficient asbestos in fine fibres is shaken up with water, and the whole poured over the broken glass. In this way the asbestos is evenly distributed on the top, and forms a suitable filtering bed (A). Pass a little water through to carry off loose pieces, and when well drained pour in the solution of chromic acid or other corrosive fluid (L). C is a cover to prevent evaporation.

Note 9.—It is necessary to boil well to secure solution; the acids do not chemically react, and the reaction between arsenious acid and carbonate of potassium is only partial.

Note 10.—In these preparations also a weak chemical combination occurs. The chloride of ammonium is added to Liq. Hydr. Perchlor. *not* to aid solution, but to prevent decomposition. Solution of perchloride of mercury alone gradually deposits oxychloride. Iodide of potassium in Liq. Iodi aids solution, iodine being only very sparingly soluble in water.

Note 11.—These formulæ are given according to the evident *intention* of the B. P. to order solutions containing 1 part in 100 fl. parts. The acids are added to prevent deposition of basic salts, the spirit as a preservative from fungoid growths. Sulphate of morphine does not deposit a basic salt from its solution, hence no acid is used.

Note 12.—The gum should be in *small* pieces (powder will not do, as it never forms a clear mucilage, due to changes in the gum produced by the drying required). If made in a bottle it should be laid upon its side and frequently revolved to prevent conglomeration of the particles. Strain through muslin.

Note 13.—Starch is only soluble in boiling water; it should be mixed *cold* and stirred continually whilst applying heat till a uniform jelly results.

Note 14.—The spirit is added to the powder first to prevent the water from causing the gum to go together into clots. It does not form a *true* solution; the gum merely swells up and produces a sort of jelly.

Note 15.—The B. P. directs that the oil shall be heated to about 149° C., and kept at this temperature for about fifteen minutes, allowed to cool, and filtered. One hundred parts of this filtered oil are put into a bottle capable of holding about 112 parts, raised to 82·2° C., and one part of phosphorus added, well shaken together until the phosphorus is completely dissolved. The object of the first heating is to thoroughly dry the oil, and coagulate a trace of albuminous matter which is removed by the subsequent filtration. It is necessary to heat the filtered oil to 82·2° C. as directed in order to melt the phosphorus, thereby increasing the solubility. It must be kept *well* stoppered in full bottles so as to avoid oxidation of the phosphorus by the air.

Note 16.—The best way to make syrup is to bring the water to the boil, then add the sugar, stir till dissolved, and strain through flannel or felt, and when strained add sufficient water to bring the sp. gr. to 1·330.

Filtration through Flannel.—Flannel strainers are made the same shape as conical calico strainers, they must be well washed in warm water before use; felt bags are made without seams, and are preferable but expensive.

Syrups of the B. P. strength will not keep in cold weather, the sugar slowly crystallises out; they should be made of sp. gr. 1·32 in winter. In making Syrup. Aurant. Flor. the orange-flower water is added when nearly cold to avoid loss of the essential oil.

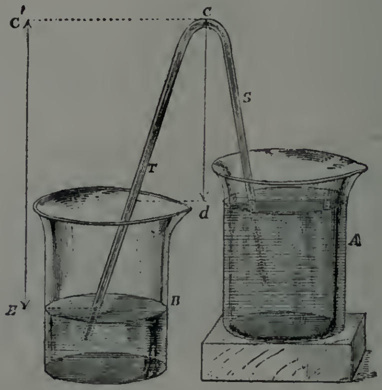
Note 17.—In the former edition of the Pharmacopœia sulphate of quinine was employed; this was far less soluble, and became cloudy by deposition of sulphate of calcium, the orange peel containing citrate of calcium. In cold weather it was also liable to throw out sulphate of quinine. These inconveniences do not now occur.

Note 18.—Equals 2 grains in 1 fl. ounce. It is well to dissolve the tartarated antimony in a little of the sherry previously heated nearly to boiling, and add the solution to the remainder of the sherry.

Note 19.—The B. P. directs that after solution the wine shall stand three days before filtration; by this means trifling deposits of tannate of iron and tannate of quinine are formed and removed. The citric acid assists solution of the sulphate of quinine, which although very sparingly soluble in water is readily dissolved by weak acid liquids.

Decantation.—There is a group of solutions which are made by a modification of this method. They are usually saturated or nearly saturated solutions of comparatively insoluble substances, and are made by repeatedly agitating the solvent with an excess of the substance, allowing the undissolved portion to subside, and removing the clear liquid by *decantation* or filtration. *Decantation* is the act of removing a clear liquid from an insoluble solid or other liquid without the use of a filter. It may be accomplished either by simply pouring off, or better by a *syphon*. The syphon consists of a tube, preferably glass, bent so that one arm is somewhat longer than the other, and similar in shape to that shown in Fig. 34. The vessel A, containing the liquid and sediment, is raised to a higher level than the receiving vessel B; the syphon S filled with some of the clear liquor (or water if permissible), the thumb or finger being pressed over the open end of the longer arm; the shorter arm is then carefully lowered into the liquid in A, keeping its open end well above the sediment. On removing the thumb from the other end the liquid flows out in a continuous stream, and can be drawn off until very little remains above the sediment. The action of the syphon is due to gravitation: the liquid in the longer arm is greater in quantity, and therefore heavier, than that in the shorter one, consequently it flows out; but of necessity draws after it, on account of the atmospheric pressure* upon the surface of the liquid in A, a further supply of liquid from the shorter arm and the vessel A. This must continue until all the liquid is drawn out or the open end of the shorter arm removed from the liquid, or until the level of the liquid in B equals that in A.

Fig. 34.



The perpendicular lines C *d* and C′ E represent the proportional force exerted by the downward pressure of liquid in the two arms of the syphon, that in the longer arm being clearly greater in the proportion of the difference between the two.

When simply pouring off a liquid it frequently runs down the outside of the dish or other vessel, due to the adhesion of the liquid to the surface of the vessel; this may be obviated by placing a glass rod against the edge from which it is poured, or by very slightly greasing the edge of the vessel with lard or soft paraffin.

The following are prepared in this manner:

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* The ordinary atmospheric pressure is about 15 lbs. upon each square inch of surface, but varies slightly according to the dryness or otherwise of the air; this pressure is caused by the force of gravity acting upon the atmosphere, or in other words is equal to the weight of the superincumbent air.

Name of preparation.	Formula.	Sp. gr.	Remarks.
Aqua Camphoræ	Camphor 1 part, distilled water 320 fl. parts	—	See note 1.
Collodion*	Pyroxylin 1 part, ether 36 fl. parts, s. v. r. 12 fl. parts	·784 (about)	See note 2.
Collodion Vesicans	Pyroxylin 1 part, blistering liquid 20 fl. parts	·933 (about)	See note 3.
Extractum Pareiræ Liquidum	Extract of pareira 1 part, distilled water and rectified spirit (3 to 1) q.s. to make 4 fl. parts	1·058 (about)	See note 4.
Liquor Calcis*	Slaked lime 2 parts, distilled water q. s.	—	= 10 grs. CaO in 1 pint. Note 5
Liquor Calcis Chlorinatæ	Chlorinated lime 1 part, distilled water 10 parts	About 1·055	Note 6
Liquor Calcis Saccharatus	Slaked lime 1 part, sugar 2 parts, distilled water 20 parts	1·052	= 7·1 grs. CaO in 1 fl. oz. Note 7
Liquor Gutta Percha	Gutta percha 1 part, chloroform 8 fl. parts, carbonate of lead, in fine powder, 1 part	—	See note 8.

Note 1.—The camphor being lighter than water is enclosed in a muslin bag, and kept at the bottom of the vessel by attaching a glass rod to the bag. After two days the liquid may be poured off for use. It contains about 1 in 1000.

Note 2.—Mix ether and spirit, and add pyroxylin. Tie in the cork, set bottle on its side, and turn over from time to time. When dissolved allow sediment to subside for several days, and decant clear liquid. Pyroxylin is not soluble in spirit alone.

Note 3.—This is too thick a preparation; half the amount of pyroxylin is sufficient.

Note 4.—Mix the spirit and water in the proportion of one of former to three of latter by measure; rub the extract smooth with a little of the mixture, then add enough to make up to measure. Allow to stand for some days, and syphon off clear liquid; filter the bottoms through paper with pumice used in the same way as with silica (p. 72). This method of preparation is adopted because it yields a more uniform product than that made direct from the root, as in B. P. 1867.

Note 5.—The B. P. directs that the slaked lime shall be washed with water until the washings no longer yield a turbidity with solution of nitrate of silver and nitric acid, that is—till free from chloride (of calcium or sodium). The undissolved portion is well shaken with 80 parts of distilled water, then allowed to subside and decanted.

Note 6.—Triturate in mortar till smooth, then shake in bottle occasionally during three hours. Finally settle, decant, and filter liquid through calico. Contains chlorinated lime equivalent to at least 2 per cent. of *available* chlorine, *i. e.* liberated by acids and available for bleaching purposes; carbonate, &c., remain insoluble.

Note 7.—Mix lime and sugar, add these to the water, and agitate in closed bottle occasionally during several hours. Finally allow to settle, and syphon off clear liquid. It should not be exposed to the air any more than can be avoided, as the lime combines with carbonic acid, which weakens and renders turbid the preparation. The sugar assists solution of the lime by forming a weak compound. Arthur has shown that if the lime contains iron this solution gradually acquires a brown coloration.

Note 8.—The carbonate of lead is only added to cause the undissolved portion of gutta percha to settle; it draws it down mechanically by its own weight, but requires several weeks to effectually clear the liquid.

Questions on Chapter V.

1. What do you understand by the terms *solution, soluble, saturated, solvent?*

2. Distinguish between *simple* solution and *chemical* solution.

3. What is specific gravity? What is the hydrostatic balance, and how is it used?

4. Describe fully the method of determining sp. gr. by the bottle.

5. What is meant by spirit of wine 33° O. P.? How much absolute alcohol do the official rectified and proof spirits contain?

6. Remark upon the following preparations:—Linimentum Opii, Linimentum Crotonis, Liquor Plumbi Subacetatis Dilutus, Liquor Ammonii Acetatis.

7. How would you prepare Acidum Sulphuricum Dilutum, Liquor Ammonii Citratis, Liquor Trinitrinæ?

8. Explain the rise of temperature attending the solution of Calcium chloride.

9. What are *filtration* and *decantation?* Name the commoner filtering media. Describe the filtration of camphorated oil.

10. Describe the preparation of an asbestos filter.

11. Describe the preparation of Glycerinum Aluminis, Glycerinum Amyli, Liquor Arsenicalis, Liquor Cocainæ Hydrochloratis, Mucilago Acaciæ, Oleum Phosphoratum, Syrupus Scillæ, Liquor Calcis.

12. Explain the object of the following:—Camphor water in Inj. Apomorph.; glycerine in Lin. Iodi; chloride of ammonium in Liq. Hydr. Perchlor.; iodide of potassium in Liq. Iodi; acetic acid in Liq. Morph. Acet.; carbonate of lead in Liq. Gutta Percha.

13. Two liquids are given you of sp. gr. 1·053 and ·878 respectively: in what proportions must they be mixed in order to produce a liquid of sp. gr. ·983?

14. Describe a water-bath.

CHAPTER VI

SOLUTION (*continued*)

Before proceeding to the notice of *chemical* solutions we must refer to a large class of preparations most of which are prepared essentially by trituration, and in which a portion of the solid ingredients is dissolved and a portion remains insoluble.

To this class belong most of the confections, cataplasms, mixtures, enemata, and pill-masses, and some of the liniments of the Pharmacopœia. These various groups of preparations vary greatly in consistency, the mixtures, enemata, and some of the liniments being liquid ; the cataplasms and confections are soft semi-solid compounds ; whilst the pill-masses are tough solids, possessing only sufficient plasticity to allow of their being rolled, cut, and rounded into pills.

The simplest of these are the confections and cataplasms, which are mere mixtures : the former are made of such consistency that they can easily be taken in a spoon ; the latter are of about the same stiffness, but are used only for external application.

Excipient.—The general rule for preparing confections is to reduce the solid ingredients to fine powder, mix them intimately, and finally add the syrup or other *excipient*, as it is called. An *excipient* is a substance added to the active ingredients of a confection, pill-mass, &c., to bring the whole to a suitable consistency.

Official confections, &c. ; those marked * to be made :

Name of preparation.	Formula.	Remarks.
Confectio Opii	Compound powder of opium 1 part, syrup 3 parts	Equals 1 of opium in 40.
Confectio Piperis	Black pepper 2 parts, caraway fruit 3 parts, clarified honey 15 parts	Equals 1 in 10.

Name of preparation.	Formula.	Remarks.
Confectio Rosæ Caninæ*	Hips, deprived of seed-like fruits, 1 part; sugar 2 parts	Equals 1 in 3. See note 1.
Confectio Rosæ Gallicæ	Fresh red rose petals 1 part, sugar 3 parts	Equals 1 in 4. Pulp the rose leaves.
Confectio Scammonii	Resin of scammony 48 parts, ginger 24 parts, oil of caraway 2 fl. parts, oil of cloves 1 fl. part, syrup 48 fl. parts, clarified honey 24 parts	Equals 1 in 3⅓ nearly.
Confectio Sennæ	—	See "Evaporation."
Confectio Sulphuris	Sublimed sulphur 4 parts, acid tartrate of potassium 1 part, syrup of orange peel 4 fl. parts, tragacanth $\frac{1}{24}$ part	Equals 1 in 2½ nearly. Note 2.
Confectio Terebinthinæ	Oil of turpentine 1 fl. part, liquorice root 1 part, clarified honey 2 parts	Equals 1 in 4.
Mel Boracis	Borax 2 parts, glycerine 1 part, clarified honey 16 parts	Equals 1 in 9½.
Cataplasma Lini*	Linseed meal 2 parts, boiling water 5	Equals 1 in 3½. Note 3.
Cataplasma Sinapis	Linseed meal 2½ parts, mustard 2½ parts, boiling water q.s.	Equals about 1 in 6. Note 4.
Cataplasma Sodæ Chlorinatæ	Solution of chlorinated soda 1 fl. part, linseed meal 2 parts, boiling water 4 fl. parts	Equals 1 in 7.
Cataplasma Fermenti	Beer yeast 3 fl. parts, wheaten flour 7 parts, water at 37·8° C. 3 fl. parts	Equals 1 in 4⅓. Note 5.

Note 1.—To remove the hard outer portion the hips are well beaten in a stone mortar, and the pulpy mass rubbed through a sieve; after repeating the operation with the portion which will not pass through, the sugar is added and well mixed. The hips alone would be too soft, and liable to fermentation and mould.

Note 2.—The tragacanth is added to give consistency to the otherwise too liquid product. This preparation is liable to dry up and become crystalline upon the surface; a little glycerine to replace some of the syrup would be an improvement.

Poultices.—*Note* 3.—The B. P. in this as other poultices orders the boiling water in fluid parts; it is, however, far better to weigh it, as glass measures would be broken, and metal ones expanded by the hot water: moreover, in a preparation the temperature of which is important, it is well not to entail the use of more vessels than are really needed. The best plan to make a poultice is to set on about double the required amount of water to boil; meanwhile the basin in which it is made is weighed, the muslin cloths prepared, and the linseed meal weighed on paper. When boiling, heat the basin with some of the boiling water, throw this away, and having placed the basin on the scales, weigh into it the boiling water. Now sprinkle in the linseed meal, continually stirring, and when mixed quickly introduce into the muslin bag or cloth.

Note 4.—Mix the mustard with about its own weight of lukewarm water; make a *soft* poultice with the linseed meal and boiling water, and add the former to the latter, stirring well. The mustard loses less of its pungent volatile oil when first made into a paste with nearly cold water as directed, than if mixed with nearly boiling water when dry.

Note 5.—Mix the yeast with the water, and stir in the flour. Place near the fire till it rises. The water must not be *hot* or the yeast would be killed; when made as described the temperature of the mixture is about 21° C., a most favorable one for fermentation, which is the cause of the *rising*, carbonic acid gas being produced. Its value as a gentle sedative is due to this gas.

Emulsions.—The liquid preparations of this class are mostly more or less perfect (or imperfect) examples of what are known as *emulsions*. An *emulsion* is a preparation consisting of a watery liquid throughout which an insoluble substance, usually an oil or resin, is evenly distributed by the aid of some third substance called the *emulsifying agent*. An emulsion cannot be considered *perfect* unless the suspension of the insoluble matter is permanent, and unaffected by dilution with water; in any case, however, a vigorous shake should cause the ingredients to mix evenly, so that a truly representative dose of the mixture can be poured out. The most important of the emulsifying agents are gums and mucilages, such as acacia, ghatti, and the natural gums present in many drugs, *e. g.* myrrh; yolk of egg; dilute spirituous preparations of drugs containing saponin, viz. tinctures of quillaia and senega; soap, and alkalies, such as Liquor Potassæ and Liquor Ammoniæ. The following list of official preparations gives several illustrations of emulsification.

Preparations marked * to be made by the student.

Name of preparation.	Formula.	Remarks.
Linimentum Ammoniæ	Solution of ammonia 1 fl. part, olive oil 3 fl. parts	Equals 1 in 4. Note 1.
Linimentum Calcis*	Solution of lime 1 fl. part, olive oil 1 fl. part	Equals 1 in 2. Note 1.
Linimentum Hydrargyri	Ointment of mercury 1 part, solution of ammonia 1 fl. part, camphor liniment 1 fl. part	Equals 1 in 3 nearly. Note 2.
Linimentum Terebinthinæ*	Soft soap 2 parts, distilled water 2 parts, camphor 1 part, oil of turpentine 16 fl. parts	Equals 1 in 10½. Note 3.

Name of preparation.	Formula.	Remarks.
Enema Aloës	Aloes 40 grains, potass. carbonate 15 grains, mucilage of starch 10 fl. oz.	Equals 1 in 111. Note 4.
Enema Asafœtidæ	Asafœtida 30 grains, distilled water 4 fl. oz.	Equals 1 in 60 nearly. Note 5.
Enema Magnesii Sulphatis	Sulphate of magnesium 1 oz., olive oil 1 fl. oz., mucilage of starch 15 fl. oz.	Equals 1 in 17 nearly. Note 6.
Enema Terebinthinæ	Oil of turpentine 1 fl. oz., mucilage of starch 15 fl. oz.	Equals 1 in 16.
Mistura Ammoniaci*	Ammoniacum, in coarse powder, 1 part; distilled water 32 fl. parts	Equals 1 in 32 nearly. Note 5.
Mistura Amygdalæ	Compound powder of almonds 1 part, distilled water 8 fl. parts	Equals 1 in 8 nearly. Note 7.
Mistura Creasoti	Creasote 1 fl. part, glac. acetic acid 1 fl. part, spirit of juniper 2 fl. parts, syrup 32 fl. parts, distilled water 480 fl. parts	Equals 1 in 516. Note 8.
Mistura Cretæ	Prepared chalk 1 part, gum acacia, in powder, 1 part; syrup 2 fl. parts, cinnamon water 30 fl. parts	Equals 1 in 34½ nearly. Note 9.
Mistura Ferri Composita	Sulphate of iron 1 part, carbonate of potassium 1⅕ parts, myrrh and refined sugar, of each 2⅖ parts; spirit of nutmeg 9 fl. parts, rose water 170 fl. parts	Equals 1 in 186. Note 10.
Mistura Guaiaci	Guaiacum resin 1 part, sugar 1 part, gum acacia, powdered, ½ part; cinnamon water 40 fl. parts	Equals 1 in 42 nearly. Note 11.
Mistura Olei Ricini*	Castor oil 180 fl. parts, oil of lemon 5 fl. parts, oil of cloves 1 fl. part, syrup 45 fl. parts, sol. potash 30 fl. parts, orange-flower water to make 480 fl. parts	Equals 3 in 8 by measure. Note 12.
Mistura Scammonii	Scammony 1 part, milk 146 fl. parts	Equals 1 in 147. Note 13.
Mistura Spiritûs Vini Gallici	French brandy 4 fl. oz., cinnamon water 4 fl. oz.; yolks of 2 eggs, sugar ½ oz.	Equals 1 in 2½ nearly. Note 14.
Tinct. Chloroformi et Morphinæ	Chloroform 109 fl. parts, ether 27¼ fl. parts, s. v. r. 109 fl. parts, hydrochlorate of morphine 2 parts, diluted hydrocyanic acid 54½ fl. parts, oil of peppermint 1 fl. part (nearly), liquid extract liquorice 109 fl. parts, treacle 109 fl. parts, syrup q. s. to 875 fl. parts	Equals 1 grain of hydrochlorate of morphine in 1 fl. oz. Note 15.
Vapor Olei Pini Sylvestris	Fir-wool oil 40 fl. parts, light carbonate of magnesia 22 parts, water to produce 480 fl. parts	Equals 1 in 12. Note 16.

Note 1.—This is the simplest possible form of an emulsion; the two are simply shaken together in a bottle until the mixture is thin enough to pour out. The object of the oil in Lin. Ammoniæ is as a *lubricant*.

Note 2.—Mix Liq. Ammon. with half the Lin. Camph., rub the Ung.

Hydrarg. with the other half, and mix them together. The result is an unsatisfactory preparation.

Note 3.—Mix the soap and water to a smooth consistence; dissolve camphor in oil of turpentine, then rub these together until thoroughly mixed, adding the turpentine gradually. The preparation is almost a jelly, but the addition of two parts of water renders it fluid and nearly white. It is then a good emulsion.

Note 4.—The carbonate of potassium assists the solution of the aloes. Rice starch should not be used for the mucilage.

Note 5.—**Gum-resins in Mixtures.**—Asafœtida and ammoniacum contain resin, essential oil, and gum. For the enema and mixture firm whitish pieces should be selected, as containing most gum, and rubbed to a powder in a mortar. At first only sufficient water is added to form a stiff paste, with which the drug is rubbed *smooth*, then the remainder of the water added gradually, finally straining through muslin. The water dissolves the gum, which effects the emulsification of the resin and oil. Preparations of this character should never be made from the finely *powdered* gum-resin, but fresh pieces should be powdered as required for use, because it is necessary to drive off some of the oil before reducing to fine powder, or it will soon become a mass again.

Note 6.—As shown by Ince, the B. P. directions give a poor result, the following method of mixing being satisfactory. Dissolve the salt in half the water, make a mucilage of starch with the other half, with which incorporate the oil; finally add by degrees the solution of magnesium sulphate.

Note 7.—Made like Mist. Ammoniaci. The gum acacia aids in emulsifying the oil of almonds.

Note 8.—Mix creasote and acid; gradually add water, and lastly syrup and spirit of juniper. This is not an emulsion, merely a solution.

Note 9.—This is not an emulsion; the chalk and gum are mixed in a mortar, the cinnamon water added gradually, and lastly the syrup.

Note 10.—Pick pieces of myrrh, which when broken show many whitish streaks of gum; reduce these to powder, rub with the carbonate of potassium and sugar, and just sufficient rose water to form a paste (not too thin), mixing *thoroughly;* now add the rose water gradually and the spirit of nutmeg. The sulphate of iron is added immediately before dispensing, for if kept all together, the mixture, which should be of a greenish colour, becomes brown, owing to the action of the oxygen of the air. The gum of the myrrh together with the potassium carbonate effect an imperfect emulsification of the resin of the myrrh. When freshly prepared it contains ferrous carbonate, chemical action occurring between the sulphate of iron and carbonate of potassium.

Note 11.—The gum acacia emulsifies the guaiacum resin, but not perfectly.

Note 12.*—**Castor Oil Emulsion.**—The B. P. formula and directions are both very unsatisfactory. It makes a fair emulsion if mixed as follows: —Mix the oils in the mortar, and add the syrup; mix the solution of potash with the orange-flower water, and add gradually to the oils and syrup. A far better emulsion is made by mixing castor oil two parts with powdered acacia one part in a mortar, adding all at once two parts of distilled water and triturating till thoroughly mixed, then adding more water gradually if required. Any flavouring may be used; if an oil it should be mixed with the castor oil at first; but if aqueous or spirituous it should be mixed with the last portion of the water. Much spirit would destroy the emulsion by coagulating the gum.

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* See Conroy, 'C. and D.,' January 3rd, 1891.

Note 13.—Scammony contains resin and gum. The milk assists emulsification; it should be made as required, for an obvious reason.

Note 14.—The yolk of egg is not needed to form an emulsion; it is admitted as a nutrient. Rub the yolks with sugar, then add half the cinnamon water, and finally the other half mixed with the brandy.

Note 15.—Mix the Morph. Hydrochl., spirit, oil of peppermint, chloroform, and ether. Mix the liquid extract of liquorice with the treacle and about 330 fl. parts of the syrup, and add this to the spirituous liquid. Mix thoroughly by agitation in a bottle, add the hydrocyanic acid, and finally the remainder of the syrup.

Note 16.—Rub the oil with the carbonate of magnesium and add the water gradually. The magnesia serves to keep the oil in a finely divided state.

These examples serve to bring under notice several methods of emulsification. It is usually safest to prepare emulsions by trituration in a mortar, but the expert dispenser can produce equally good preparations of a great many oily and resinous drugs by simple agitation in a bottle.

Emulsions by Trituration.—This is the method adopted in all cases where it is required to produce an emulsion of gum-resin—that is, a drug containing both a resin and a gum, *e. g.* Enema Asafœtidæ, Mistura Ammoniaci, &c.; or when an oil, &c., is emulsified by the aid of powdered gum or yolk of egg; and in many cases in which mucilage of acacia, tragacanth, ghatti, starch, or Irish moss or soap is employed. Instances of these have in some cases been described, *e.g.* ammoniacum mixture, castor oil emulsion, liniment of turpentine. Many other emulsions may be made on similar lines, such as cod-liver oil or extract of male fern with powdered acacia; cod-liver oil or castor oil with yolk of egg and tragacanth. When yolk of egg is used it is first thoroughly triturated with a little oil, water added as it gets thick, then more oil and water alternately with diligent trituration.

Cod-liver oil emulsion.—Cod-liver oil emulsion may be made most satisfactorily by trituration with powdered acacia, or ghatti, or mucilage of Irish moss. For the emulsion with gum proceed as follows:—Three fluid ounces of cod-liver oil are mixed with the flavouring oils according to taste, the most suitable being almonds, orange, lemon, and cinnamon. The mixture is added to 6 drachms of finely powdered acacia, or one third that quantity of ghatti, contained in a mortar; 10 fl. drs. of water are now added all at once, and the whole briskly stirred until a white cream results; sufficient water is now added, mixed with the syrup, glycerine, elixir of saccharin, or other sweetening agent required, to produce 6 fl. oz. of product. For an Irish moss emulsion ¼ oz. of moss is rinsed with cold water, then digested with 24 fl. oz. of water for one hour, and boiled for five minutes. The resulting mucilage is strained through muslin, and then cleared by filtration through cotton wool. To 2 fl. oz. of this mucilage 3 fl. oz. of cod-liver oil are gradually added with brisk trituration, the flavours being introduced afterwards, and the whole made up to measure with distilled water.

Emulsions by Agitation.—No pharmacist would ever think of preparing such emulsions as liniment of ammonia or lime by trituration, they may be so readily mixed by simple agitation. The method is, in fact, usually adopted when alkalies, such as solution of potash or lime, and tinctures of senega or quillaia are the emulsifying agents; and also frequently with mucilage, especially for such emulsions as those of very resinous tinctures, such as guaiacum or cannabis indica.

Copaiba emulsion.—For copaiba, which may be taken as an example, proceed as follows:—Into a 6-oz. bottle introduce 3 fl. drs. of solution of potash and 2 fl. oz. of water; now pour in carefully 6 fl. drs. of copaiba without touching the side or neck of bottle; agitate *briskly*, and dilute to about 5 fl. oz. with water added in divided portions, agitating between each; add now any spirit of nitre or other spirituous preparations, and finally make up to measure with water.

Tincture of guaiacum.—For tincture of guaiacum or cannabis proceed similarly. Dilute 6 fl. drs. mucilage of acacia with 2 fl. oz. of water, shake up so as to wet sides of bottle, pour in 6 fl. drs. of the tincture in small quantities, agitating briskly; lastly dilute with water in portions to make 6 fl. oz.

Or an emulsion may be made by triturating 1 fl. oz. of the tincture with 1 oz. of yolk of egg, and dilute as usual.

Emulsions with tincture of senega or quillaia are made similarly, ½ fl. dr. of tincture being sufficient for 1 fl. oz. of castor or cod-liver oil, according to Collier.

The student should practise the preparation of emulsions by the various methods until he has become expert in the art. When large quantities are made, an apparatus on the principle of the common egg-whisk is of great value.

Lin. Potass. Iodidi c̄ Sapone.—There is one preparation which may be mentioned here, being prepared partly by solution and partly by trituration ; it is scarcely an emulsion, being of rather a more solid consistence : this is liniment of iodide of potassium and soap. "Take of curd soap 16 parts, iodide of potassium 12 parts, glycerine 8 fl. parts, oil of lemons 1 fl. part, distilled water 80 fl. parts. Reduce the soap to fine shreds, and dissolve this in the water and glycerine upon the water-bath. When dissolved pour into a mortar in which the iodide of potassium has been previously powdered. Mix briskly, and continue trituration until cold. Set aside an hour, then rub well the oil of lemon to a cream-like product." It is allowed to stand an hour to permit the escape of some of the entangled air-bubbles, so as to avoid as much as possible the action of the air on the oil of lemon.

We now pass to a consideration of pill-masses and pills ; all the official pill-masses come under this section except three, viz. Pil. Ferri Iodidi, Pil. Scammonii Comp., and Pil. Ferri.

Pills.—A pill-mass consists of two parts ; the medicine proper, or active ingredient, and another substance which is added to give the requisite consistency, and called the *excipient.* If the active medicine be a solid, the excipient is usually a liquid or semi-solid ; if it be a liquid, the excipient will be a solid or semi-solid. The choice of a suitable excipient is a great part of the art of pill-making ; it should not be capable of dissolving the whole of the active ingredients, but it should either dissolve a small portion, or it should contain some adhesive substance, such as gum, sugar, &c. ; in fact, in all cases where the active ingredients do not *contain* some gum or similar adhesive body, it is distinctly advantageous, and in most cases necessary, that the *excipient shall do so.* On this account it is important that the operator should know precisely the properties and composition of the drugs upon which he works; he will then seldom find difficulty in making pills. The object aimed at by every dispenser is to produce a mass which possesses sufficient firmness to prevent the pills from losing their shape when kept, and also sufficient plasticity to permit of ready manipulation. When the drugs employed are of an insoluble or sparingly soluble nature it is necessary that this plasticity shall be permanent, or they will in all probability pass unchanged through the system. It is scarcely necessary to point out that these qualities of firmness and plasticity are mainly dependent upon the insolubility and solubility of the active ingredients in the excipient or *vice versâ*; hence the need of *partial* solubility above noted.

Rule for Pill-making.—The general rule to be observed in preparing a pill-mass is as follows:—*Thoroughly mix the dry ingredients by powdering and trituration, then add the excipient and beat or work into a mass.* The excipient should be added all at once; the amount can be judged for any given mass after practice. If liquid it may be measured or dropped from a glass rod. It is usual to make the pill-mass in a mortar; the working up is accomplished by using a long, thin pestle, and pressing hard upon the contents of the mortar, using the side of the mortar as a fulcrum for the lever.* When massed it is weighed into suitable quantities, and *rolled* and *cut* upon the pill machine. The pill machine consists of two parts, one of which rests upon the working bench; the other is held in the hands. The latter consists of a hard wooden board about 3 to 4 inches wide, and having handles at each end; one side of this board is perfectly flat, the other has screwed on to it a brass plate having hemispherical grooves running across in the narrow direction: the number of grooves is commonly twenty-four, and their width and depth vary according to the sized pills the machine is intended to make, which is usually 1, 2, 3, 4, or 5 grains. The former part of the machine likewise consists of two portions, a flat, hard wood or marble slab, which is arranged nearest to the operator, and beyond this a brass plate bearing hemispherical grooves exactly coinciding with those in the other part; the whole is enclosed in metal bands to prevent warping of the wood, and these bands project to some extent above the surface of the wooden plate. The mass is rolled into a more or less spherical form, placed on the flat part of the machine, and rolled with the palm of the hand to a short cylinder, then with the flat side of the second part of the machine it is rolled out until it has extended into a long cylinder or *pipe* of the required length; it must be noted whether this is of equal thickness throughout during the operation, and if not pressure must be so applied that the inaccuracies may be corrected. When so *piped* the cylinder is put across the metal grooves, the handle turned over, and by a careful pressure and rolling motion, given by the wrist and hand, it is cut into the desired number of more or less perfect pills. These are again removed to the flat board, and *finished* or *rounded* by means of a level disc with a raised rim, with which they are covered and rapidly revolved, using suitable pressure according to the hardness of the pills.

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* Small quantities may, however, be made with a spatula upon a porcelain slab.

Dusting Powder.—For these operations some *dusting powder* is generally needed to prevent the pills from sticking to each other or the machine; the commonest powders are various kinds of starch, especially arrowroot, alone or mixed with French chalk, lycopodium, powdered liquorice or althæa, and magnesium carbonate. A little dusting powder is also put into the box in which they are kept.

Excipients.—The excipients in use are very numerous; almost every dispenser has certain favourites, to which, on account of long practice, he pins his faith. The most important are water, syrup, treacle, glucose syrup, mucilage of acacia, confection of rose, confection of hips, spirit, glycerine, glycerine of tragacanth, castor oil, oil of theobroma, soap, soft paraffin, crumb of bread, powdered tragacanth, powdered liquorice and other woody drugs, sugar of milk, &c. This list might be multiplied almost indefinitely, and many of these are used in various combinations, *e. g.* a mixture of glycerine with mucilage of acacia is very commonly employed. It is not intended to enter into detail as to the use of all these excipients, for which the student should consult a work on dispensing, or Mr. Ince's admirable paper on pills in 'Pharmaceutical Journal' [3], xv, pp. 1009, 1027, but a few general remarks will assist him in choosing the most suitable for most purposes.

Water is only used as an excipient when the active drugs contain a gum or some other soluble adhesive substance in sufficient quantity; it must be used cautiously, or it will produce very soft pills.

Syrup, treacle, glucose syrup.—These are more adhesive than water in the order given, and may be used when that quality is lacking, *e. g.* for many inorganic and organic chemicals, such as sulphate of iron, and for woody drugs like rhubarb, ginger, &c.

Mucilage of acacia.—Used in similar cases to syrup, but the pills are liable to become hard and insoluble.

Confection of hips, confection of roses.—Possess a fair degree of adhesiveness, but a larger quantity is required; used especially for chemicals, aloes, and similar drugs. Confection of roses should not be used for alkaloids, which would form insoluble tannates with the tannin it contains.

Spirit.—Must be used with great caution, in small quantity only; it should only be used when a resinous drug like scammony or jalapin is combined with *much* woody matter, such as rhubarb or althæa, or in conjunction with soap. A small quantity is often advantageously combined with a large proportion of some other excipient, such as syrup.

Glycerine.—This also must be used with caution, and seldom without some other adhesive excipient, such as mucilage or syrup. If used in too large a quantity it not only makes the pills soft, but after they have been made they absorb moisture from the air and give the appearance of sweating. It is, however, of great value in preventing the undue hardening of pills containing woody drugs.

Glycerine of tragacanth.—This is a most useful excipient; it possesses the properties of glycerine to a modified degree, the tragacanth tending to solidity. It is the excipient *par excellence* for such chemicals as quinine, strychnia, arsenious acid, &c. For many of these substances which are given in fractions of a grain it may be conveniently combined with sugar of milk, in such quantity as to make the finished pill weigh 1 *grain*, as suggested by Mr. Martindale. Pills so made are sufficiently firm, but retain their plastic condition for a long time.

Castor oil.—Very seldom used; is ordered in B. P. for Pil. Hydrarg. Subchlor. Composita.

Oil of theobroma, or white wax.—These may be used for essential oils in small doses, or for phosphorus; the active substance is dissolved in the oil or wax after melting, and when nearly cold it is rolled quickly into pills.

Soap.—Is commonly used for resins and many liquids, such as creasote and volatile oils; it is often combined with more adhesive excipients.

Soft paraffin.—This, combined with some other similar substances, which are not readily oxidised, may be advantageously used for such powerful chemicals as permanganate of potassium, oxide or nitrate of silver, &c. A useful mixture is Mr. Martindale's "kaolin ointment,"—soft paraffin 1, hard paraffin 1, kaolin 1.

Crumb of bread.—Should be one day old; used for creasote, oil of savin, &c.

Powdered tragacanth.—Gives toughness to a mass; a small quantity will frequently prevent "falling." It should be mixed with the powder. It acts by forming a strong mucilage with any water which is present.

Powdered liquorice, althæa, &c.—Give solidity; used for many liquids, and to impart firmness to unstable masses.

Pill-coating.—The subject of pill-coating is treated in Chapter XXIV.

The following table gives the pill-masses of the Pharmacopœia, classified according to the excipients used.

Name of pill-mass.	Active ingredients.	Excipient.	Remarks.
Pilula Aloës Barbadensis	B. aloes 16 parts, hard soap, in powder, 8 parts; oil of caraway 1 fl. part	Confection of roses 8 parts	1 in 2 nearly.
Pilula Aloës et Asafœtidæ	S. aloes 1 part, asafœtida 1 part, powdered hard soap, 1 part	Confection of roses about 1 part or q. s.	About 1 in 3½; less than 1 of treacle needed.

Name of pill-mass.	Active ingredients.	Excipient.	Remarks.
Pilula Aloës et Ferri	Sulphate of iron 1½ parts, B. aloes 2 parts, compound powder of cinnamon 3 parts	Confection of roses 4 parts	Glycerine and p. tragacanth preferable; 1 of aloes in 5½ nearly.
Pilula Aloës Socotrinæ	S. aloes 16 parts, hard soap, in powder, 8 parts, volatile oil of nutmeg 1 fl. part	Confection of roses 8 parts	1 in 2 nearly.
Pilula Ferri Carbonatis	Saccharated carbonate of iron 4 parts	Confection of roses 1 part	1 in 1¼.
Pilula Plumbi cum Opio	Acetate of lead, in fine powder, 6 pts.; opium, in powder, 1 pt.	Confection of roses 1 part	1 of opium in 8.
Pilula Hydrargyri	Mercury 2 parts	Confection of roses 3 parts, liquorice root, in fine powder, 1 part	1 of mercury in 3. Note 1.
Pilula Asafœtidæ Composita	Asafœtida 1 part, galbanum 1 part, myrrh 1 part	Treacle 1 part	1 in 4. Note 2.
Pilula Conii Composita	Extract of conium 5 parts, ipecacuanha, in powder, 1 part	Treacle q. s.	Excipient unnecessary.
Pilula Ipecacuanhæ cum Scillâ	Compound powder of ipecacuanha 3 parts, squill, in powder, 1 part; ammoniacum 1 part	Treacle q. s.	About 1 of opium in 20.
Pilula Scillæ Composita	Squill, in powder, 1¼ parts; ginger, in powder, 1 part; ammoniacum, in powder, 1 pt.; hard soap, in powder, 1 part	Treacle 2 parts or q. s.	1 in 5.
Pilula Aloës et Myrrhæ	S. aloes 2 parts; myrrh 1 part, saffron, dried, ½ part	Treacle 1 part, glycerine q. s.	About 1 in 2½.
Pilula Rhei Composita	Rhubarb 6 parts, S. aloes 4½ parts, myrrh 3 parts, hard soap 3 parts, all in powder; oil of peppermint ⅓ part	Glycerine 2 parts, treacle about 6 parts	About 1 in 4. Requires only 1½ glycerine and 4½ treacle.
Pilula Saponis Composita	Opium, in powder, 1 part; hard soap, in powder, 4 parts	Glycerine q. s.	1 of opium in 6 nearly.
Pilula Ferri	Sulphate of iron 120 parts, carbonate of potassium 72 parts	Sugar 24 parts, tragacanth 8 parts, glycerine 4½ fl. parts, water q. s.	About 1 of carbonate of iron in 5.
Pilula Ferri Iodidi	Iodine 80 parts, iron wire 40 parts	Sugar 70 parts, liquorice root, in powder, 140 parts; water 46 fl. parts	See p. 95. = 1 of FeI_2 in 3½.
Pilula Colocynthidis Composita	Colocynth pulp 4 parts, B. aloes 8 parts, resin of scammony 8 parts, sulphate of potassium 1 part, all in powder; oil of cloves 1 fl. part	Distilled water q. s.	1 in 6 nearly. Mucilage of tragacanth preferable.
Pilula Colocynthidis et Hyoscyami	Compound colocynth pill 2 parts, extract of henbane 1 part	—	1 of colocynth in 8½ nearly.
Pilula Cambogiæ Composita	Gamboge, in powder, 1 part; B. aloes, in powder, 1 part; compound powder of cinnamon 1 part; hard soap, in powder, 2 parts	Syrup q. s.	1 in 6 about. Glycerine of tragacanth preferable as excipient

Name of pill-mass.	Active ingredients.	Excipient.	Remarks.
Pilula Hydrargyri Subchloridi Composita	Subchloride of mercury 1 part, sulphurated antimony 1 part, guaiacum resin, in powder, 2 parts	Castor oil 1 fl. part or q. s.	1 in 5 about. Preferably mucilage of acacia $\frac{2}{3}$ part, glycerine $\frac{1}{3}$.
Pilula Phosphori	Phosphorus 1 part	Balsam of tolu 40 parts, yellow wax 19 parts, curd soap 30 parts	1 in 90. Note 3.
Pilula Scammonii Composita	Resin of scammony 1 part, resin of jalap 1 part, curd soap, in powder, 1 part; strong tincture of ginger 1 fl. part	Rectified spirit 2 fl. parts	1 of res. scam. in $3\frac{1}{4}$.

Soap has been included amongst the "active" ingredients; as a matter of fact, it is partly used to promote the action of the purgative, partly as an excipient for resinous substances. The student should prepare at least six of the above.

Note 1.—The mercury is rubbed with confection till no globules are visible, the liquorice added and thoroughly mixed.

Note 2.—The B. P. directs heat to be used to melt the gum resins; it is preferable to reduce them to powder, whilst keeping cold, and mass with glycerine in a warm mortar.

Note 3.—The phosphorus is melted beneath hot water, and thoroughly incorporated with the balsam of tolu beneath the water at about 60° C.; the wax added and mixed. When required for use, two parts of this mass are mixed with one part of soap, with a little rectified spirit, if necessary.

Questions on Chapter VI.

1. Define *excipient*, *emulsion*, *confection*.

2. Describe the preparation of confection of rose, mustard poultice.

3. What is the emulsifying agent in each of the following preparations?—Mistura Olei Ricini, B. P.; Linimentum Terebinthinæ; Enema Asafœtidæ; Mistura Amygdalæ; Mistura Guaiaci.

4. Describe the preparation of an emulsion of (1) tincture of guaiacum; (2) castor oil, using gum acacia; and (3) copaiba.

5. Describe the preparation of Mist. Ferri Composita.

6. Give the special characters, as pill excipients, of mucilage of acacia, glycerine, spirit, tragacanth, and soft paraffin.

7. Comment upon the B. P. formulæ for Pilula Colocynthidis Comp., Pil. Rhei Comp., Pil. Asafœtidæ Comp., Pil. Conii Comp., and Pil. Hydrarg. Subchlor. Comp.

8. What temperatures are officially directed to be employed in preparing (1) Pilula Phosphori, (2) Cataplasma Fermenti, and why?

CHAPTER VII

SOLUTION (*continued*)

Group 3.—*Solutions dependent upon Chemical Action.*

Some of the solutions mentioned in Chapter V are nearly allied to these, *e.g.* Liq. Calcis Saccharatus; but in this group is included only those cases of solution in which marked chemical change occurs, *e.g.:*

Liquor Ammonii Citratis fortior (*to be made by student*).

"Take of—

Citric acid	12 parts.
Strong solution of ammonia	11 fl. parts, or a sufficiency.
Distilled water	a sufficiency.

"Neutralise the acid with the ammonia, adding sufficient distilled water to yield twenty-four fluid parts of product."—B. P.

The ammonia should be added gradually to the acid, to which a small quantity (about 4 fl. parts) of the water has been previously added, keeping the whole cool by immersing the containing flask in cold water. The point of neutrality is determined by means of litmus papers; when really neutral a red paper will become faintly blue on immersion in the liquid and rinsing with distilled water.

There are a great many preparations and chemicals whose production is attended by solution dependent upon chemical action; by far the greater number, however, are not simple processes of solution, but involve other processes in addition. Those which involve chemical solution, accompanied only by processes already noticed, are included in the following tables, the reactions which occur being expressed by chemical equations.

Preparations marked * to be made by the student.

Name of preparation.	Formula.	Equation.	Sp. gr.	Per cent. active ingredient.	Note
Liquor Ammonii Acetatis fortior	Carbonate of ammonium 15½ parts, acetic acid 50 fl. parts, or q. s. to neutralise, distilled water sufficient to produce 60 fl. parts	$NH_4HCO_3,NH_2COONH_4 + 3CH_3COOH = 3CH_3COONH_4 + 2CO_2 + OH_2$	1·071	35·5 per cent.	1 and 2
Liquor Ammonii Citratis fortior	Citric acid 12 parts, strong ammonia solution 11 fl. parts, or q. s. to neutralise, distilled water to produce 24 fl. parts	$3NH_4HO + H_3C_6H_5O_7\dagger = (NH_4)_3C_6H_5O_7 + 3OH_2$	1·209	47·6 per cent.	2
Liquor Arsenii et Hydrargyri Iodidi	Iodide of arsenic 1 part, red iodide of mercury 1 part, distilled water to produce 100 fl. parts	$AsI_3 + HgI_2 = AsI_3,HgI_2$	1·016	1 per cent. each AsI_3 and HgI_2	3
*Liquor Bismuthi et Ammonii Citratis	Citrate of bismuth 1 part, solution of ammonia q. s. to dissolve, distilled water to produce 10·9 fl. parts	$BiC_6H_5O_7 + 2NH_4HO = BiO(NH_4)_2C_6H_5O_7 + OH_2$	1·070	8·54 per cent. citrate = about 3 grs. Bi_2O_3 in 1 fl. dr.	4
Liquor Ferri Pernitratis	Iron wire 1 part, nitric acid 4½ fl. parts, distilled water to produce 30 fl. parts	$Fe_2 + 8HNO_3 = Fe_2 6NO_3 + 2NO + 4OH_2$	1·107	13·1 per cent. pernitrate = 2·6 grs. Fe_2O_3 per fl. dr.	5
Liquor Hydrargyri Nitratis Acidus	Mercury 4 parts, nitric acid 5 fl. parts, distilled water to produce 12 parts (weight)	$3Hg + 8HNO_3 = 3Hg 2NO_3 + 2NO + 4OH_2$	About 2·00	54 per cent. mercuric nitrate	6
Liquor Magnesii Citratis	Carbonate of magnesium 2½ parts, citric acid 5 parts, syrup of lemons 5½ fl. parts, bicarbonate of potassium 1 part, water to produce 110 fl. parts	$3(MgCO_3)_3,Mg(HO)_2 + 8H_3C_6H_5O_7 = 4Mg_3,2C_6H_5O_7 + 9CO_2 + 15OH_2$ $3KHCO_3 + H_3C_6H_5O_7 = K_3C_6H_5O_7 + 3CO_2 + 3OH_2$	—	3·5 per cent magnesium citrate = 10 grs. mag. carb. per fl. oz.	7
*Liquor Plumbi Subacetatis	Acetate of lead 5 parts, oxide of lead, in powder, 3½ parts, distilled water to produce 20 fl. parts	$\left.\begin{matrix}CH_3COO\\CH_3COO\end{matrix}\right\} Pb + PbO = \left.\begin{matrix}CH_3COO\\CH_3COO\end{matrix}\right\} Pb_2O$	1·275	24 per cent. $Pb_2O(C_2H_3O_2)_2$	8

Liquor Potassæ	Carbonate of potassium 1 part, slaked lime ¾ part, distilled water 10 fl. parts	$K_2CO_3 + Ca(HO)_2 = 2KHO + CaCO_3$	1·058	5·84 per cent. KHO	9
Liquor Sodæ	Carbonate of sodium 1¾ parts, slaked lime ¾ part, distilled water 10 fl. parts	$Na_2CO_3 + Ca(HO)_2 = 2NaHO + CaCO_3$	1·017	4·1 per cent. NaHO	9
Liquor Sodæ Chlorinatæ	Chlorinated lime 1 part, carbonate of sodium 1½ parts, distilled water 10 fl. parts	$Na_2CO_3 + Ca(ClO)_2 = 2NaClO + CaCO_3$ $Na_2CO_3 + CaCl_2 = 2NaCl + CaCO_3$	1·054	2·5 per cent. Cl as hypochlorite	10
Liquor Sodii Ethylatis	Sodium, free from oxide, 1 part, ethylic alcohol 20 fl. parts	$2C_2H_5HO + Na_2 = 2C_2H_5NaO + H_2$	0·867	19 per cent. C_2H_5NaO	11
*Liquor Strychninæ Hydrochloratis	Strychnia 1 part, diluted hydrochloric acid 1½ fl. parts, rectified spirit 24 fl. parts, distilled water 73 fl. parts	$C_{21}H_{22}N_2O_2 + HCl = C_{21}H_{22}N_2O_2,HCl$	0·978	1 part strychnine in 100 fl. parts	12
Oleatum Hydrargyri	Yellow oxide of mercury 1 part, oleic acid 9 parts	$2HC_{18}H_{33}O_2 + HgO = Hg(C_{18}H_{33}O_2)_2 + OH_2$	—	10 per cent. mercuric oxide	13
Oleatum Zinci	Oxide of zinc 1 part, oleic acid 9 parts	$2HC_{18}H_{33}O_2 + ZnO = Zn(C_{18}H_{33}O_2)_2 + OH_2$	—	10 per cent. zinc oxide	14
*Syrupus Ferri Iodidi	Iron wire 1 part, iodine 2 parts, refined sugar 28 parts, distilled water 13 fl. parts	$Fe_2 + 2I_2 = 2FeI_2$	About 1·385	5·7 per cent. FeI_2 = 4·3 grs. per fl. dr.	15
Syrupus Ferri Subchloridi	Iron wire 3 parts, hydrochloric acid 8¾ fl. parts, citric acid $\frac{1}{10}$ part, distilled water 5½ fl. parts; syrup to produce 87½ fl. parts	$Fe_2 + 4HCl = 2FeCl_2 + 2H_2$	About 1·340	4·82 per cent. $FeCl_2$ = 3·55 grs. per fl. dr.	16
Tinctura Quininæ Ammoniata	Sulphate of quinine 1 part, solution of ammonia 7 fl. parts (nearly), proof spirit 48 fl. parts	$([C_{20}H_{24}N_2O_2]_2H_2SO_4)_2,15OH_2 + 4NH_4HO =$ $2(NH_4)_2SO_4 + 4C_{20}H_{24}N_2O_2,3H_2O + 7OH_2$	·9335	2 per cent. sulphate of quinine (nearly) = 1 gr. per fl. dr.	17
Pilula Ferri Iodidi	See page 91	$Fe_2 + 2I_2 = 2FeI_2$	—	28 per cent. nearly = 1 gr. FeI_2 in 3½ grs.	18

† Constitutional formula (p. 9), $COOH,CH_2,C(OH)(COOH),CH_2,COOH$.

Note 1.—Crush ammon. carb.; add acetic acid gradually; when nearly all has been added, test for neutrality from time to time as follows:—Take out a few drops of the solution in a watch-glass, and add a drop of acetate of lead solution: if a white precipitate appears, more acetic acid is needed; if no precipitate, warm a little of the solution in a test-tube by immersion in boiling water, agitate to assist escape of carbonic acid gas and test with a blue litmus paper; it should not become decidedly red. When this point is reached, filter through paper, and wash the filter with water till the required measure is obtained.

Note 2.—Neither of these solutions can be kept in bottles containing *lead*, as that metal is dissolved by them from the glass.

Note 3.—Triturate the iodides with a small proportion of the water, filter, and wash with water to produce required volume. Iodide of mercury is insoluble in water, but it forms a soluble double iodide with iodide of arsenic; a little metallic arsenic usually remains undissolved. (Donovan's solution.)

Note 4.—Rub the citrate of bismuth to a paste with a little water, add the solution of ammonia gradually, stirring continually until the salt is just dissolved. Dilute with about half the water, filter through paper, and wash the filter with the water till the required volume is produced. This solution does not keep well; the use of 10 per cent. of rectified spirit, or of 5 per cent. of each glycerine and rectified spirit in place of some of the water, will prevent the fungoid growth from forming. The equation given above is suggested as the probable chemical reaction.

Note 5.—Dilute the acid with sixteen parts of water, and in this dissolve the iron wire, moderating the action by keeping cool; when all the iron is dissolved, filter, and make up to 30 fl. parts with distilled water. If the action be allowed to proceed too rapidly a dark-coloured liquid is obtained, or even a part of the iron refuses to dissolve, but remains as an insoluble oxy-salt; this is due to the unnecessary loss of nitric acid by elimination as N_2O_3 or N_2O_4 instead of NO.

Note 6.—Mix the nitric acid and water in a flask, and dissolve the mercury without heat. Boil gently for fifteen minutes, cool, and preserve the solution, which should weigh about twelve parts, in a stoppered bottle, away from the light. The boiling expels nitric oxide, which otherwise remains dissolved.

Note 7.—See also Chap. VIII. The magnesia is dissolved in about twenty parts of water by aid of the acid, and filtered into a soda-water bottle; syrup added, and enough water to nearly fill the bottle; finally the bicarbonate of potassium; then securely corked, and tied or wired over. When opened effervescence occurs, owing to the escape of carbon dioxide.

Note 8.—**Dilution to a given sp. gr.**—Boil all together with constant stirring for half an hour, or until the litharge has dissolved. The equation given expresses the reaction in the simplest form; it is, however, not very definite. The solution is allowed to cool, decanted or syphoned from any insoluble matter, the bottoms filtered through a double paper filter, and diluted with water to correspond with the specific gravity required. To find the amount of water needed, take the sp. gr. of the filtered liquid, from this subtract the sp. gr. required, then the amount by which the required sp. gr. exceeds 1000 will represent the quantity of strong solution to be taken, and the difference between the required and found sp. gr. will be the quantity of water needed. An example will make this clear.

Sp. gr. of decanted solution (55 fl. oz.)	1·300	Sp. gr. required	1·275
Sp. gr. required	1·275	Water . .	1·000
	25		275

To every 275 fl. parts of solution (sp. gr. 1·300) 25 fl. parts of water must be added to produce 300 fl. parts of solution of sp. gr. 1·275, *i. e.* every 11 require 1, consequently 5 fl. oz. will be wanted.

Note 9.—Boil together in a clean iron or silver vessel till the liquid is free from carbonate. This is ascertained by allowing the carbonate of calcium to settle, removing a small quantity of the clear liquid, and adding an equal bulk of lime water; if perfectly free from carbonate it will remain clear. The clear liquor is removed by a syphon, adjusted to the official sp. gr., and kept in a well-stoppered bottle to prevent absorption of carbon dioxide from the air. An iron or silver vessel is used because these metals are not acted upon by potash or soda solution of this strength. Solution of potash may also be prepared in a green glass bottle without heat, if sufficient time be allowed (twelve hours or so), and frequently shaken. Slaked lime, although soluble in water, is insoluble in solution of potash or soda of official strength, hence the absence of lime from these preparations.

Note 10.—Dissolve the carbonate of sodium in 2½ parts of the water, triturate the chlorinated lime with the remainder until smooth, and filter. Mix the two solutions and again filter. Calico is the best filtering medium. Must be kept in a cool, dark place, as light and heat accelerate its change into chloride and chlorate of sodium: $3(NaCl,NaClO) = 5NaCl + NaClO_3$.

Note 11.—The sodium must be clean. The whole must be kept cool during solution. If kept, the solution becomes discoloured, and carbonate of sodium is gradually deposited by action of air.

Note 12.—This is intended as a 1 per cent. solution. The acid and one third of the water are mixed, and the strychnine dissolved by raising to boiling. The alkaloid strychnine is nearly insoluble, but its hydrochlorate dissolves readily. The amount of acid ordered in the B. P. is in excess of that required to produce the hydrochlorate, and the alternative formula in parts contains even more acid. The result is the production of the acid hydrochlorate, which is only very slightly soluble in water or water and spirit, consequently in cold weather the liquid deposits crystals of that salt. It would be more stable if prepared with 1¼ fl. parts of diluted hydrochloric acid (*vide* P. J. [3], xxii, p. 843).

Note 13.—Solution takes place readily in the cold if the oxides be added gradually and the whole well stirred. The 20 per cent. oleate keeps better, and may be prepared similarly (1 of HgO to 4 of oleic acid), or more rapidly by the application of a temperature of 38° to 49° C.

Note 14.—Stir the oxide into the oleic acid, allow to stand two hours, then heat gently until the oxide is dissolved.

Note 15.—(1) Prepare a syrup by dissolving the sugar in 10 parts of the water. (2) Put the iron and the iodine in a flask with the remaining 3 parts of water, and gently warm till action commences; moderate the reaction by cooling if necessary; when nearly complete, heat to near boiling and agitate till the froth becomes white; now add 2 fl. parts of the syrup and boil for ten minutes, then filter the liquid into the remainder of the syrup (using a "capped" filter) and mix. Wash the filter with sufficient water to produce 43 parts of syrup. The object of boiling with part of the syrup is to convert some of the cane-sugar into dextrose, which is said to have a preserving influence upon the syrup. The addition of ½ per cent. of hypophosphorous acid (30 per cent.) is more effectual. Syrup of iodide of iron should be stored in *full* bottles, in a good light, with a small coil of clean iron wire in each; this materially retards the discoloration which occurs, and renders such additions unnecessary. For the *pill-mass*, the iron, iodine, and water, are allowed to act as for syrup, and when finished the liquid is poured over the sugar, triturated, and finally mixed with the liquorice.

Note 16.—Mix the HCl with about 4 parts of the water, add the wire, and heat until action ceases. Add the citric acid, and filter through paper into about 40 fl. parts of the syrup; then wash the filter with the remainder of the water. Add sufficient syrup to make up to the required volume. The citric acid preserves the ferrous chloride from oxidation.

Note 17.—Diffuse the sulphate of quinine through the spirit and add the solution of ammonia. Shake till dissolved; no heat is needed. Filter, if necessary, through paper or cotton wool.

Questions on Chapter VII.

1. Describe the preparation of Liquor Arsenici et Hydrargyri Iodidi; Liquor Plumbi Subacetatis; Liquor Sodii Ethylatis; Syrupus Ferri Iodidi.

2. Give chemical equations illustrating the preparation of Liquor Ammonii Citratis fortior; Liq. Ferri Pernitratis; Liquor Sodæ; Liquor Hydrargyri Nitratis Acidus, and Liq. Sodæ Chlorinatæ.

3. Give the percentage of active ingredient in Liquor Potassæ; Liquor Sodæ; Pilula Ferri Iodidi; Syrupus Ferri Subchloridi; Tinct. Quininæ Ammon.; Liq. Strychninæ Hydrochloratis.

4. Give notes on the preservation of Liq. Ammonii Acetatis; Liquor Bismuthi et Ammonii Citratis; Liquor Potassæ; Liquor Sodæ Chlorinatæ; Liquor Sodii Ethylatis; Liquor Strychninæ Hydrochloratis; Syrupus Ferri Iodidi.

5. What are the objects of the following steps in processes?—(1) Boiling for fifteen minutes when making Liq. Hydrarg. Nitrat. Acid.; and (2) boiling with part of the syrup for Syr. Ferri Iodidi.

6. How much carbonate of sodium will be required to produce 1 litre and and 300 cubic centimetres of solution of soda (B. P.)?

7. How much iron, calculated as metal, is contained in 1 pint of the official syrup of iodide of iron, assuming that no iodine is lost in the process? Express the result in grains, and also in the fraction of a kilogramme.

CHAPTER VIII

SOLUTION (*continued*)

Group 4.—Solution of Gases.

Solution of Gases.—The preparation of most of these belongs properly to the art of applied chemistry; an exception is—

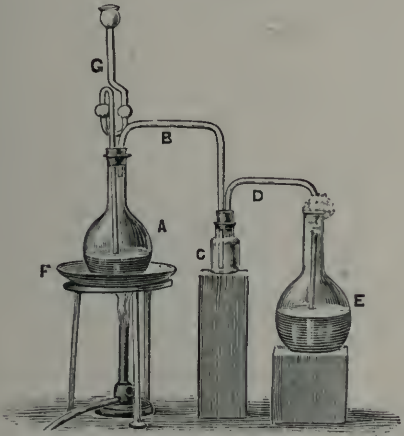
Liquor Chlori (to be made by the student).

Take of—

Hydrochloric acid	6 fl. parts.
Black oxide of manganese	1 part.
Distilled water	34 fl. parts.

The oxide, hydrochloric acid, and two parts of water are put into flask A (Fig. 35), which is connected by tube B with the small bottle C, containing 2 parts of water (to wash the gas from acid, &c., accidentally carried over); thence by

FIG. 35.



tube D to flask E, containing 30 fl. parts of water, and loosely plugged with tow or wool. Heat is applied to A by means of a sand-bath F until gas ceases to be evolved; the flask E is then corked and shaken to dissolve the chlorine. A safety-tube, G, is also connected with the flask A, to allow air to pass in, so that the liquid in C should not return when Cl. ceases to be evolved.

Chlorine water must be kept cool and dark, as heat and light promote the following reaction :—$2Cl_2 + 2H_2O = 4HCl + O_2$.

Glass tubes may be bent by holding them in the upper part of a flat gas flame (not Bunsen flame), revolving them constantly; when quite soft they can be bent into any required shape.

The other preparations made in the same way are—

Name.	Equation.	Sp. gr.	Strength.
Acidum Hydrochloricum	$2NaCl + H_2SO_4 = Na_2SO_4 + 2HCl$	1·16	Note 1. About 32 per cent. by weight.
Acidum Sulphurosum	$4H_2SO_4 + C_2 = 4SO_2 + 2CO_2 + 4H_2O$ $SO_2 + H_2O = H_2SO_3$	1·025	Note 2. 5 per cent. SO_2 by weight.
Liquor Ammoniæ fortior	$2NH_4Cl + Ca(HO)_2 = 2NH_3 + CaCl_2 + 2H_2O$	·891	Note 3. 32·5 per cent. NH_3 by weight.
Liquor Chlori (as above)	$MnO_2 + 4HCl = MnCl_2 + 2H_2O + Cl_2$	1·003	About 2·66 grs. of Cl per fl. oz. = 0·6 per cent.

Note 1.—In all operations of solution of gases in water it must be borne in mind that gases dissolve more readily in cold water than in hot.

Note 2.—Sulphurous acid by absorption of oxygen from the air becomes converted into sulphuric acid, thus: $2H_2SO_3 + O_2 = 2H_2SO_4$.

Note 3.—Strong ammonia solution must be kept cool and well stoppered, or it will lose strength by evolution of NH_3. The ordinary commercial article is stronger than the official; its sp. gr. is ·880.

Diluted hydrobromic acid (B. P.) is a solution of hydrobromic acid gas in water, but its method of preparation being entirely different it is described elsewhere. The same remark applies in a certain sense to diluted hydrocyanic acid, hydrocyanic acid (HCN) being gaseous at 26·5° C.

Aërated Waters.—There is also a distinct class of preparations whose production depends upon the solution of a gas, viz. *aërated waters.* In these, however, the *gas* is not the active principle, although doubtless its presence modifies the action of the medicine employed; it is used to impart pleasantness and brilliancy to the preparation, and in some cases also to effect the solution of the active substance. The gas almost universally employed is carbonic acid gas, although oxygen has been occasionally used for special cases. The official aërated solutions will be first mentioned, after which a short description of their manufacture will follow.

Name of preparation.	Formula.	Equation.
Liquor Lithiæ Effervescens	Carbonate of lithium 10 grs., water 1 pint	$L_2CO_3 + CO_2 + H_2O = 2LHCO_3$. (See Note 1.)
Liquor Magnesii Carbonatis	Sulphate of magnesium 2 pts., carbonate of sodium 2½ pts., distilled water q.s. See also "Precipitation"	$4MgSO_4 + 4Na_2CO_3 + H_2O = 3MgCO_3,Mg2HO + 4Na_2SO_4 + CO_2$. $3MgCO_3,Mg2HO + 5CO_2 + 3H_2O = 4(MgCO_3H_2CO_3)$. (See Note 2.)
Liquor Magnesii Citratis	See page 94	
Liquor Potassæ Effervescens	Bicarbonate of potassium 30 grs., water 1 pint	None. (See Note 1.)
Liquor Sodæ Effervescens	Bicarbonate of sodium 30 grs., water 1 pint	None. (See Note 1.)

Note 1.—Commonly known as lithia, potass, and soda water, but the last mentioned is seldom made of B. P. strength unless as "*medicinal* soda water."

Note 2.—Commonly known as "fluid magnesia;" second equation given as probable explanation of solution of carbonate by CO_2 and water.

There are a great many medicinal and other aërated beverages, the most important of which are—

Seltzer water, containing chiefly chlorides of magnesium, sodium, and calcium.

Aërated lime water.—Liquor Calcis (B. P.) aërated.

Lemonade.—A sweet beverage acidulated with citric acid and flavoured with tincture of fresh lemon peel.

Ginger beer.—Like lemonade, but flavoured with ginger.

Ginger ale.—An aromatic beverage containing capsicum, ginger, and various flavours, less sweet than ginger beer.

Solubility of Gases.—The production of an aërated beverage depends upon the fact that gases dissolve in water more freely under high pressure than at low pressure; when, therefore, the pressure is removed by drawing the cork, some of the gas escapes, giving the sparkling effect. This solubility follows a fixed law, viz. *the solubility of a gas in water or other liquid varies directly as the pressure; e. g.* carbonic acid gas is soluble under ordinary atmospheric pressure and at 15·5° C. to the extent of about 1 in 1 of water by volume; if, however, the pressure be doubled the solubility will be 2 in 1, and so on.

Manufacture of Aërated Drinks.—The apparatus required are: (1) a "generator" for production of the CO_2; (2) a "washer" for the CO_2; (3) a "gasometer" for storing the CO_2 previous to use; (4) a "cylinder" in which the beverage is aërated; and (5) various "filling machines."

The *generator* consists of a hard-wood vat which is furnished with stirrers worked by machinery, and closely covered to prevent escape of gas, which is conducted by a strong pipe to the "washer" or "scrubber." The gas is produced by the action of diluted sulphuric acid on whiting, chalk, or bicarbonate of sodium, the acid being introduced gradually to avoid its too rapid disengagement. The washer or *scrubber* contains water, or preferably permanganate of potassium solution, through which the gas bubbles and thereby becomes freed from traces of acid mechanically carried over, or from traces of nitrous acid existing as impurity in the sulphuric acid used, or of sulphuretted hydrogen from the presence of sulphide in the chalk. After washing, the gas passes into the "gasometer," which consists of a large inverted metal tub—in fact, a miniature gas-holder such as are used for illuminating-gas works. From the gasometer a pipe carries the gas to the *cylinder*, which consists of a strong copper vessel with tin lining, having an inlet through which the water may be pumped, and another for the gas, which is also forced in by pumping. In this way, the water and gas being pumped in simultaneously, a strong solution of the gas is obtained, the strength of which may be gauged by means of a pressure gauge attached to the cylinder. An outlet pipe carries the solution of gas (aërated water) to the "filling machine." The cylinder also has a waste tap at the bottom, for running off unused water at the end of the day. The bottles having been cleaned, and a sufficient quantity of a strong solution of bicarbonate of sodium (or other medicament or syrup required) having been measured into each, they are now brought to the filling machine, by which each is filled up with the aërated water and corked. These machines are of many patterns, and it will serve no useful purpose to describe the various kinds ; it will suffice to say that by opening a certain valve the pressure from the cylinder causes the aërated water to fill the bottle, which is then at once corked by the descent of a cylindrical metallic rod, and passed on to be "*wired*." This is done by means of thin iron or copper wire, whereby the cork is secured. In larger quantities it is usual either to employ a machine which shall automatically measure the required amount of strong solution, or to make the solution of the strength as bottled, and pump this into the cylinder with the gas instead of making plain aërated water first.

The most important points to be considered in making aërated preparations are—

1. *Scrupulous cleanliness*, not only of bottles, but of all machinery and apparatus, by frequent washing.

2. *Thorough filtration* of all solutions and syrups, and the water itself.

3. *Freedom from impurity, especially lead*, to be attained by using only pure chemicals for the actual beverages ; washing the gas, and having all parts of the machinery which the water comes in contact with, either of pure tin or of tin-lined copper, or silver-plated.

4. *Careful method* in all the work, so that all goods of one kind are finished off and labelled before any possibility occurs of mistakes arising.

Liquor Magnesii Citratis is prepared differently, the gas being produced in the bottle itself by the action of citric acid on bicarbonate of potassium.

Questions on Chapter VIII.

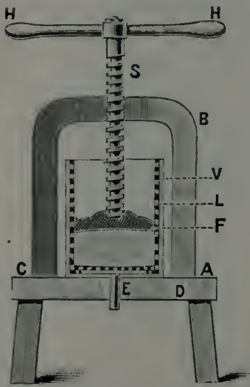
1. Describe the preparation of sulphurous acid, with equation.
2. Gives notes on the preservation of Liquor Chlori and Liq. Ammon. fort.
3. What is the relation between the solubility of gases and the pressure and temperature ?
4. Give the strength of Liquor Sodæ Effervescens, Liq. Lithiæ Effervescens, and Liq. Magnesii Carbonatis.

CHAPTER IX

EXPRESSION

PRESSURE is constantly resorted to in pharmacy for the purpose of removing watery or other liquids from solids. This operation of squeezing one substance *out of* a crude drug or mixture is usually called *expression.* It forms a secondary part of the process in the manufacture of most of the tinctures, many fluid extracts, extracts, wines, &c., and is the principal operation in the preparation of the official and other juices, and fixed oils.

FIG. 36.



Screw Press.—The presses employed for pharmaceutical purposes are of two kinds, screw and hydraulic; the former is the kind commonly used in retail pharmacies, the latter being suited for large quantities. Fig. 36 represents a form of screw press in section. The screw S, turned by the handles H H, passes through a thick guiding framework A B C, of wrought iron, which is securely bolted to the wooden block D, supported at a convenient height upon four legs which are screwed to the floor. The "box" into which the substance to be pressed is placed consists of a tinned iron cylindrical vessel V, the sides and bottom of which are about $\frac{1}{8}$ to $\frac{1}{3}$ inch thick according to the size of the press; this box is provided with an exit spout, E, for the expressed liquid, which is sometimes situated in the centre of the bottom, at others in the side. This box contains a porous lining L, also of tinned iron (or tinned copper), the bottom and sides being separate to facilitate cleaning, the disc which forms the bottom being raised about $\frac{1}{8}$ to $\frac{1}{4}$ inch above the bottom of the "box" by a number of projections upon its under side. The thick moveable lid F passes freely into the porous lining, and has a depression in the centre of its upper side to receive the end of the screw. The substance to be pressed is enveloped in "cheese-cloth," muslin, calico, or canvas, according to its nature and quantity; cheese-cloth is the most generally useful, but finely divided chemicals like phosphate of iron must be pressed in fine calico or linen. Such finely divided substances are tied up tightly in the cloth and then introduced into the press, but in most other cases the press box is lined with the cloth, the drug introduced, and the ends of the cloth neatly and evenly turned in to cover the surface; the box is returned to the press, the lid put in, and the screw *gradually* brought to bear with its full force, a jar or other receiver having been placed under the spout. *Too rapid application of pressure will often result in bursting the cloth.*

To determine the total pressure produced on the top plate of the press box we require to know (1) the power applied to the handles = P; (2) the length of the handles between the points at which P is applied = H; and (3) the pitch of the screw, that is the depth from any point in the thread to the same point in that part of the thread directly below it = S. These data being known, the effective pressure L exerted on the plate will be found as follows:

P × the distance through which it travels =
L × " " "

It is obvious that L travels through the distance S, and P through the distance H × 3·1416, because H represents the *diameter* of a circle. Therefore $LS = P \times H \times 3{\cdot}1416$,

$$\text{or } L = \frac{P \times H \times 3{\cdot}1416.}{S}$$

To take an example, let P = 60 lbs.
H = 20 inches.
S = ½ inch.

$$\text{Then } L = \frac{60 \times 20 \times 3{\cdot}1416}{\frac{1}{2}}.$$

$$L = 7540 \text{ lbs.}$$

This allows nothing for loss by *friction*, which in a press working well would amount to about 25 per cent.; this would reduce the effective pressure to 7540 – 1885 = 5655 lbs. If it be required to ascertain the pressure on each square inch of surface, this total (5655 lbs.) must be *divided* by the area of the plate expressed in square inches, A. Supposing this to be 50 square inches, then the pressure per square inch = 113·1 lbs.

The Hydraulic Press.—The *hydraulic press*, which is alone suitable for the expression of fixed oils from seeds, &c., involves an entirely different principle, viz. that *a fluid in a closed vessel perfectly transmits through its whole substance whatever pressure is applied to any part.* For example, suppose we have a closed metal box, the total inner surface of which is 1000 square inches, and connected with this a tube having a section of 1 square inch; both box and tube are filled with water, and a pressure of 1 lb. applied to the surface of the liquid in the tube; this pressure is transmitted to every portion of the inner surface of the box, and *every square inch* of the surface of the box will receive a pressure of 1 lb., or a total pressure of 1000 lbs. upon the entire surface.

Fig. 37.

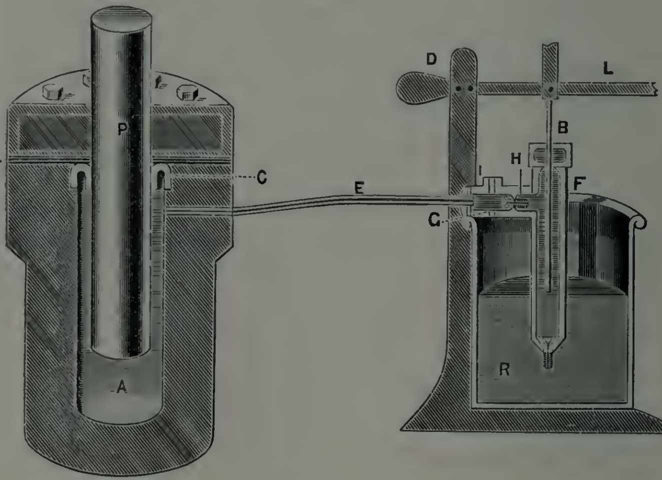


Fig. 37 represents the hydraulic press in section. It consists of a suction and force-pump F, worked by means of a lever L, turning about the axis D. B is the piston. The water is drawn from the reservoir R, and forced along the tube E into the cistern A. Fitting into this cistern is a heavy plunger P, leakage being prevented by the leather collar C which surrounds the plunger. Above the plunger, which is also called the *ram*, and attached to it, is a strong plate or block, called the *follower*, on which is placed the substance to be pressed, either in a box similar to that used in the screw press, or packed in strong cloths arranged in layers having metal or hard-wood plates or boards between each, with an arrangement for collecting and carrying off the expressed liquid; the pressure is exerted against a rigid framework above. This framework and press box are not shown in the figure. As the pump is worked and the cistern A becomes filled, the plunger rises and transmits pressure to the contents of the cloths or press box. The forcing pump is furnished with a safety-valve covering the pipe I, the pressure being controlled by a weight attached to this valve; it also possesses a valve G for removing the pressure by allowing the water to flow backward from the cistern A into the reservoir R. H is a valve admitting water to the tube E from R, but resisting its return.

The amount of pressure obtained depends upon the ratio of the section of the plunger P to the section of the piston B, and upon the length of the lever used to work the pump. Suppose the length of the lever = 30 inches, the distance between the fulcrum D and the piston B is 3 inches, and the power applied to L is 50 lbs., then by the principle of the lever the force exerted upon the piston will be $\frac{50 \times 30}{3} =$ 500 lbs. Let the section of plunger be 100 times that of the piston B, then the total force exerted upon the plunger P will be $500 \times 100 = 50{,}000$ lbs.

Succi.*—THE JUICES of the Pharmacopœia are of two kinds, those which are pressed and used at once for the manufacture of other preparations, and those which are preserved by the addition of rectified spirit. To the former class belong lemon and mulberry juices, which appear in the B. P. under the names of the respective fruits; the other class, which consists of the juices of belladonna, conium, henbane, broom, and taraxacum, being arranged under the general term Succus. At least one of these must be prepared by the student. The B. P. directions for the preparation of the "succi" are alike, as follows:

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* For seasons of collection see Calendar, p. 21.

Succus Belladonnæ.

Take of—

Fresh leaves and young branches of belladonna	7 lbs.
Rectified spirit	a sufficiency.

Bruise the belladonna in a stone mortar, press out the juice, and to every three measures of juice add one of the spirit. Set aside for seven days, and filter. Keep it in a cool place.

In the case of henbane, flowering tops are also used; broom, the fresh tops, with or without accompanying leaves; taraxacum, the fresh root.

On the large scale bruising in a stone mortar is replaced by passing the drug through rollers, or by crushing in an ordinary stone drug-mill. The "mush" so produced is pressed in canvas, or, on the small scale, in cheese-cloth. The addition of the spirit slowly causes some of the mucilaginous matter present in the juice to become insoluble; hence the necessity for setting aside during a week or more. That which cannot be decanted clear should be filtered through paper pulp as follows:

Take several sheets of stout filtering paper, tear into small pieces, and put into a jar or bucket. Add now sufficient boiling water to cover them, stir and beat thoroughly with a suitable stirrer until the paper is quite broken up and "pulpy," throw on a muslin strainer, allow to drain, and press off the superfluous water with the hands. Mix sufficient of the "pulp" with enough of the juice to fill a conical calico filter, and pour into the filter, returning the first portions. The paper pulp will gradually form a filtering-bed on the calico, and as soon as the liquid passes quite bright it may be collected for use. Many liquids which obstinately refuse to filter bright through paper may be cleared in this way. Lemon juice must be pressed *very gradually*, or the cloth will certainly break.

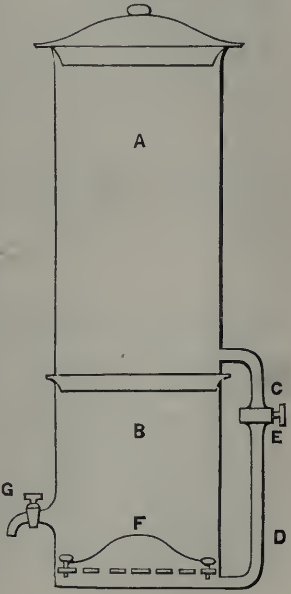
Fixed Oils.—Fixed oils may be described as greasy substances composed of compounds of oleic, stearic, or similar acids with glycerine, containing also in most cases more or less of the free acids; they are decomposed by heat.

The manufacture of fixed oils requires very great pressure, and is not usually attempted in the pharmaceutical laboratory. The official fixed oils are: Ol. Amygdalæ, Crotonis, Lini, Myristicæ Expressum, Ricini, and Theobromatis, all from the seeds; Oleum Olivæ from the ripe fruit, and Ol. Morrhuæ from the fresh cod livers. Oils of nutmeg and theobroma, being solid, are pressed while hot. Strong horsehair cloths are commonly employed. Another official oil (which is not a fixed oil) is also prepared by expression, viz. oil of lemon.

After expression the oils are set aside to deposit the greater part of the water, which is pressed out together with the oil, and then filtered.

Upward Filtration of Oils.—Perfect filtration is necessary if the oil is to keep good for any length of time; it may be carried out by means of Warner's upward filter (see Fig. 38). A is a cylindrical tinned iron vessel, with a flange rim soldered on to the bottom, of rather less diameter, and about an inch wide, so as to fit firmly into the open top of another cylindrical tin vessel of the same diameter. A tube and stopcock C is fitted into the side of A, close to the bottom, and connects at E with another tube, D, which enters B near the bottom. The filtering medium F is a cone of hat-felt projecting upward from near the bottom of the lower vessel, and secured by thumb-screws passing through the felt and two tinned iron rings into the bottom of the cylinder. The stopcock C being closed, the upper vessel is filled with the crude oil, and the stopcock C opened (all joints being properly secured), that the oil may

Fig. 38.



flow into the open space below the filter. The filtered oil, as it accumulates in B, should be drawn off by the tap G, as any large amount retards the flow by decreasing the force of the column bearing on the filter.

On the large scale *filter presses* are frequently employed. A description of these is scarcely within the scope of a work intended for students and retail pharmacists; it will be sufficient to mention the main features. Filter presses consist of a series of metal or wood plates, the rims of which are thicker than the other portions, so that when placed together chambers are enclosed between each pair of plates. These plates are usually arranged vertically, and covered with the filtering medium—linen, or swansdown and paper. The liquid to be filtered is introduced into the chambers by a pump, and passes through the cloths; it is carried off by grooves in the plates, through taps, into a trough below the press. A considerable pressure can be exerted by means of the pump, and thus the filtration becomes rapid.

Frequently oils require bleaching. This is accomplished either by filtration through animal charcoal or fuller's-earth, or by treatment with chemicals, especially sulphuric acid, bichromate of potassium and sulphuric acid, and chlorine, or in some cases simply by exposure to strong light.

Questions on Chapter IX.

1. The lever of an hydraulic press is 24 inches long, and the short arm 3 inches long; the surfaces of the two plungers are 2 square inches and 1½ square feet. A pressure of 66 lbs. is applied to the handle. What is the total pressure on the 'large plunger'?
2. Describe carefully the preparation of "Succus Hyoscyami."
3. Name the official fixed oils and their sources.
4. Why is the spirit added to the official succi?
5. The depth of thread of the screw of a hand-press is ⅔ inch; the handle is 27 inches long. What is the total pressure exerted on the box-plate if a power of 75 lbs. be applied to the end of the handle? Express the answer also in metric weights.

CHAPTER X

EVAPORATION

In pharmacy it is frequently necessary to separate a more volatile substance from a less volatile one; different terms have been applied to this operation according to the object sought. If the more volatile portion is of no value, as when watery solutions are concentrated with the object of obtaining the solid matter or less volatile liquid from the solution, the process is called *evaporation*.

Exsiccation.—When the object is to remove water or other liquid from a more or less damp solid not in solution, or from crystalline solids such as carbonate of sodium (which naturally contains water in combination), the process is called *desiccation* or *exsiccation*.

Distillation and Sublimation.—When the object is to obtain the volatile liquid or solid, the non-volatile portion being of secondary importance, evaporation is combined with subsequent cooling of the vapour produced, and the terms *distillation* and *sublimation* are applied.

Distillation, exsiccation, and sublimation will be considered in subsequent chapters.

Ebullition.—Evaporation is commonly accomplished by *ebullition* or *boiling*. Most substances when heated to a certain temperature boil, *i. e.* bubbles of vapour rise from the heated surface of the dish, and rising to the surface of the liquid the film breaks, liberating vapour into the surrounding atmosphere. This ebullition occurs at a constant temperature for each substance, provided the pressure of the atmosphere is uniform; but evaporation also takes place to a smaller extent at temperatures below the boiling-point.

Rate of Evaporation.—The rate of evaporation increases very rapidly as the temperature rises; in the case of water, for example, the proportional amount evaporated in a given time at 37·8° C., 69° C., and 100° C. is about 1, 5, aud 16 respectively. The rate of evaporation of a liquid below its boiling-point depends *directly upon its surface;* that of a boiling liquid upon the area of the *heating* surface of the pan.

Latent Heat of Steam.—If heat be applied to water the temperature of the water gradually rises till the boiling-point is reached, 100° C., when it immediately becomes stationary; and, in fact, it requires the application of heat for a considerable time before the whole of the water becomes converted into steam, during which time neither the water nor the steam registers a temperature above 100° C. by the thermometer. The sensible heat so lost is said to have become *latent, i.e.* hidden, and is spoken of as the "latent heat of steam."

If one gramme of water at 0° C. be heated to 100° C., and the amount of heat required be represented by 100° C., then the amount of heat needed to convert this gramme of water at 100° C. into steam at 100° C. will be 537° C. If now this steam be passed into sufficient cold water or be otherwise cooled it will be condensed into water, and in so doing will impart exactly 537° C. to that water before the whole will have been condensed to water at 100° C.; in fact, the *latent heat* will have again become *sensible heat.* This point will be again referred to when treating of distillation. It is, therefore, clear that by far the greater part of the heat employed in ebullition is used for converting the liquid into a gas (steam), and only a small proportion for first raising the temperature to the boiling-point. If there were no such thing as *latent* heat, evaporation at the boiling-point would be unmanageable; immediately the boiling-point was reached the whole of the liquid would suddenly pass into vapour, leaving the pan dry. Other liquids behave similarly, but the amount of heat rendered latent is usually smaller.

The boiling-point of a liquid is raised by the presence of substances in solution; a saturated solution of chloride of calcium, for example, boils at above 176° C. This fact must be remembered when evaporating solutions of salts and especially organic substances, or they will be injured by the high temperature employed.

Evaporation is usually conducted in open pans, which may be of metal, porcelain, &c.; the pans should be shallow and wide, so as to present as large a surface of liquid as possible, proportionate to the quantity; this allows of the maximum speed of evaporation. They should be so situated that the vapour may be quickly diffused by draughts, as the speed of evaporation depends also on the humidity of the surrounding atmosphere. To promote the same object the liquid should be kept in continuous motion by stirring, and the stirring should be so directed as to break the surface as much as possible without undue production of froth. Stirrers are commonly made of hard wood (box) or of glass rods. Glass rods may readily be broken at any point if previously marked round with a file; the sharp ends can be rounded by a good gas flame. Mechanical stirrers worked by steam are used in large operations; they should consist of a double set of oars or teeth, working in opposite directions, or they do not break the surface sufficiently to give good effect. Stirring also prevents solids becoming baked on the pan at the edge of the liquid.

Sources of Heat.—The pans may be heated by direct gas flame, or a clear coke furnace if the substance is not injured by a high temperature; but in other cases various baths are employed, viz. sand, steam, or water baths. The gas burner used may be one of those already described (pp. 71, 72), or any of the forms in which a number of small jets of gas are emitted from a ring.

Furnace.—The laboratory furnace is an important structure; for this and many other purposes it may be constructed of fire-brick, having an iron door in front, and the air admitted to the fire through a grating below, the waste flue being near the top, at the back. A good draught is indispensable, and should admit of regulation by means of a "damper" in the flue as well as by opening or closing the door in front. The top has rings of cast iron, to admit of vessels of various sizes being heated.

The *sand-bath* is used for operations requiring less heat; it has already been described, as has also the *water-bath*.

Steam-bath.—Steam-baths are similar to water-baths, but "the vapour of water at a temperature above 212° F. (100° C.) but not exceeding 230° F. (110° C.) is similarly applied"

(B. P.). Steam-baths present a most valuable source of heat, for by their use aqueous liquids can be boiled without fear of burning the substances in solution, and the amount of liquid evaporated is greatly in excess of that by a water-bath. Steam-pans are usually made of tinned copper or enamelled iron, and are heated by means of a "jacket" or a "coil," or both. Jacketed pans consist of a pan riveted into a slightly larger iron casing, leaving a small space between the two for the steam, which enters by a pipe direct from the boiler; at the bottom of the jacket there is a small waste-pipe to carry off the water produced by condensation of steam. The jacket should not extend under the whole surface of the evaporating pan, or "baking" will be liable to occur when the liquid is reduced below the steam level. Fig. 39 illustrates a steam boiling pan, which has a larger heating surface and much greater depth than an evaporating pan. It is well to have an outlet tap at the bottom, as shown in the figure, so that the liquid can be run out into a receiver below. Such a boiling pan is conveniently arranged on a swivel, so that the contents may be poured out, thus saving the time spent in dipping or syphoning (Fig. 40). Large pans are more rapidly heated by a "coil," which consists of a wide tinned copper tube rolled spirally *inside* the pan; but this renders effectual cleaning more difficult.

Fig. 39.

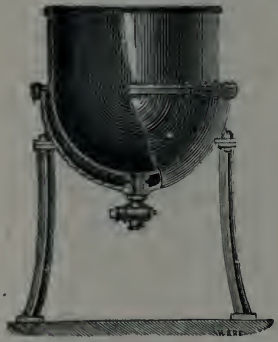
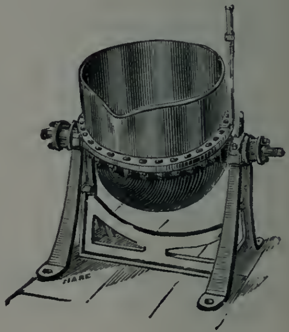


Fig. 40.

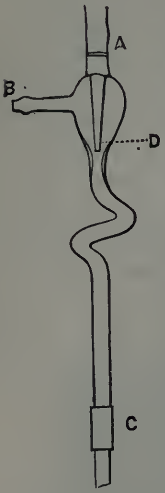


Temperature of Steam-baths.—The temperature of the steam used depends upon the pressure in the boiler; the greater the pressure the higher the boiling-point of the water, and, in consequence, the temperature of the steam also. To illustrate the influence of pressure, half fill a strong flask with water and boil, remove the flame, and tightly close the neck with a cork in which a thermometer is placed. The temperature recorded will now be slightly below 100° C. After about half a minute pour a little cold water on the upper part of the flask; immediate reduction of pressure will occur consequent upon the condensation of steam in the upper part of the flask, and the water will begin to boil rapidly. This may be repeated several times without any further application of heat; the thermometer will then register many degrees below the boiling-point of water, 100° C. If now the thermometer be raised out of the water into the aqueous vapour above, the temperature will be seen to be about the same as that of the water. The converse of this might also be shown, but the experiment is rather more likely to end in the bursting of the flask, and this one illustrates the point, viz. the boiling-point depends on the pressure. The pressure in the boiler is due to the production of steam in the boiler being greater than the outlet pipe can carry off at the ordinary pressure of atmosphere, and also to difficulty in traversing the pipes from the boiler, and may be further increased by suitable valves. The following table indicates the temperature of steam corresponding to various pressures.

Lbs. on square inch.	Temp. F.	Temp. C.	Lbs. on square inch.	Temp. F.	Temp. C.
1	102·1°	39·0°	17	219·6°	104·2
3	141·6	60·9	20	228·0	108·8
6	170·2	76·8	25	240·1	115·6
9	188·3	86·8	30	250·4	121·3
12	202·0	94·5	40	267·3	130·7
14·7	212·0	100·0	50	281·0	138·3

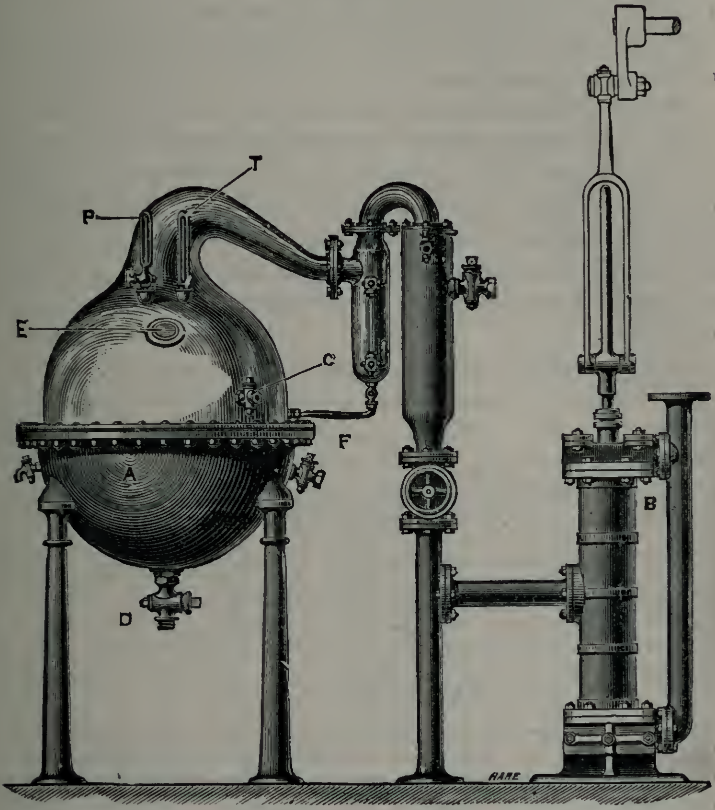
Evaporation under Reduced Pressure.—From a consideration of the above facts one is naturally led to the conclusion that evaporation under reduced pressure would be a most advantageous method in those numerous instances in which the temperature of boiling water even is injurious. The reduction of pressure necessary for evaporation may be attained by means of an aspirator or filter-pump, but it is usual to employ a vacuum pan in large laboratories. A very simple *aspirator* is illustrated in Fig. 41. The tube A, which terminates in a fine jet D, is connected with the water-supply; the side tube B with the flask or vessel containing the liquid, and C with a long metal or stout rubber tube having as direct a fall as possible. When water is passed through the apparatus the weight of the column causes it to suck vapour from B along with it, producing a partial vacuum: a warm water bath may be used as the source of heat. It is well to interpose a bottle between B and the flask, in case the water should flow back from the aspirator owing to reduction of pressure from any cause. This bottle should carry an india-rubber bung with three holes, through which pass glass tubes, one of which is connected with the aspirator, one with the flask, and the third with a long bent glass tube (30 or more inches long), the open end of which dips into a vessel of mercury; by means of this tube the degree of exhaustion can be ascertained, for the mercury rises in the tube as the pressure decreases.

Fig. 41.



Vacuum Pan.—A vacuum pan is a closed steam or water bath (Fig. 42, A), the upper part of which is drawn out into a neck and connected with an exhausting pump B, worked by steam: an engine is, of course, necessary for this pump. The steam should be applied to the pan by a coil and small jacket, or by a jacket which is divided into two parts, each of which has a separate inlet pipe for the steam; this is to avoid undue heating when the quantity in the pan is small. When properly constructed a vacuum pan is capable of boiling aqueous liquids rapidly at 40° to 50° C. The pan is fitted with pressure gauge P, thermometer T, a small pipe and tap C, through which air may be admitted when it is necessary to "break" the vacuum, and through which, by screwing on a long pipe, the liquid may be drawn into the apparatus after the air has been partly removed; an outlet tap, D, for finished product, and two sight glasses (one on each side), E, to enable the operator to see the progress of the evaporation.

FIG. 42.



Large pans are cleaned by a man who enters a manhole, but small ones have the dome to lift off by a pulley, the two being bolted together with a perfectly even flange, F. If employed for extracts or other preparations which become thick when concentrated, the vacuum pan may be provided with stirrers driven by steam power.

Evaporation to a Fixed Volume.—It is frequently required to evaporate a liquid to a given volume, and it is obviously inconvenient to transfer the hot liquid from the pan to a measure; to avoid the necessity for this, the pan should be gauged before commencing the operation by introducing the desired measure of liquid and measuring the depth. The weak liquid is then evaporated to the same depth. It is desirable to keep a record of the capacity of all fixed pans used in the laboratory at varying depths, which can then be used for reference; loose evaporating dishes are better controlled by weight.

Official Preparations.—There are no official preparations which are made simply by evaporation, but it forms an important part of the processes of producing a great number of the extracts, liquid extracts, solutions, chemicals, &c. Most of these will be considered when other processes not yet described come to be reviewed; the following, however, are made by evaporation combined with processes already described.

Those marked * should be made by the student.

Name of preparation.	Formula.	Reaction.	Notes.
Caffeinæ Citras	Caffeine 1 part, citric acid 1 part, distilled water 2 parts	$C_8H_{10}N_4O_2.OH_2 + H_3C_6H_5O_7, OH_2 = C_8H_{10}N_4O_2, H_3C_6H_5O_7 + 2OH_2$	Note 1.
Glycerinum Plumbi Subacetatis	Acetate of lead 5 parts, oxide of lead 3½ parts, glycerine 25 parts, distilled water 12 parts	$\left.\begin{array}{l}CH_3COO\\CH_3COO\end{array}\right\}Pb + PbO = \left.\begin{array}{l}CH_3COO\\CH_3COO\end{array}\right\}Pb_2O$	Note 2.
Liquor Antimonii Chloridi	Purified black antimony 1 part, hydrochloric acid 5 fl. parts, to produce 2½ fl. parts	$Sb_2S_3 + 6HCl = Sb_2Cl_6 + 3SH_2$	Note 3. Sp. gr. about 1·47.
*Liquor Ferri Perchloridi fortior	Iron wire 4 parts, hydrochloric acid 20½ fl. parts, nitric acid 1½ fl. pts., water q. s. to produce 17½ fl. parts	$Fe + 2HCl = FeCl_2 + H_2$ $6FeCl_2 + 6HCl + 2HNO_3 = 3Fe_2Cl_6 + 2NO + 4OH_2$	Note 4. Sp. gr. about 1·42.
Liquor Ferri Persulphatis	Sulphate of iron 8 parts, sulphuric acid ¾ fl. part, nitric acid ¾ fl. part, distilled water q. s. to produce 11 fl. parts	$FeSO_4 + H_2SO_4 + HNO_3 = 3Fe_2(SO_4)_3 + 2NO + 4OH_2$	Note 5. Sp. gr. 1·441.
*Oxymel Scillæ	Vinegar of squills 5 fl. parts, clarified honey 8 parts	—	Note 6. Sp. gr. 1·32.
Confectio Sennæ	Senna 7, coriander 3, figs 12, tamarinds 9, cassia pulp 9, prunes 6, extract of liquorice 1, sugar 30, distilled water to produce 75 parts	—	Note 7.

Note 1.—Dissolve the acid and the caffeine in the water and evaporate by a water-bath to dryness. Less water is preferable, thereby saving much time in evaporation. Product 92 to 93 per cent. Citrate of caffeine is a very weak compound, soluble in a very little water, but decomposed by much with liberation of caffeine.

Note 2.—"Mix and boil for a quarter of an hour, then filter and evaporate until the water is dissipated" (B. P.). Stir well during the boiling; filter warm through good paper, folded within a calico cone to strengthen it; evaporate by steam or water bath, or by sand-bath; but on no account allow the temperature to rise above 120° C., or the glycerine will be decomposed. The product should weigh about 33½ parts.

Note 3.—Dissolved by aid of heat, filtered through calico, evaporated by *boiling* to required volume.

Note 4.—Heat iron gently with 12½ fl. parts of hydrochloric acid and 7 of water, till action ceases; filter, add 7 parts of acid, and pour gradually into the nitric acid; evaporate by boiling till no more nitrous fumes are evolved and the liquid becomes cloudy (owing to separation of oxychloride); then add 1 fl. part of hydrochloric acid, and water to produce 17½ fl. parts. If the nitric acid were poured into the iron solution there would be an inconveniently violent frothing.

Note 5.—The iron solution is added to nitric acid as for perchloride, and the whole boiled down to 11 fl. parts. Enamelled iron vessels are the most suitable for these last three preparations.

Note 6.—Evaporate by a water or steam bath, and filter hot liquid through flannel in a warm place. Such filtrations may be carried out in a specially heated room or cupboard; but it is well to enclose the whole filter in a stoneware cylinder, so as to avoid evaporation and drying up of the liquid on the outside of the bag.

Note 7.—The figs and prunes are boiled with 24 fl. parts of water for four hours, tamarind and cassia pulp added, with more water to replace that lost by evaporation, digested two hours, and the pulp rubbed through a hair sieve, rejecting stones and hard parts. To the pulped product add sugar and ext. liquorice, and dissolve on water-bath; while warm sprinkle in the mixed senna and coriander powders, stirring thoroughly; finally, make the weight of product 75 parts by evaporation or addition of distilled water. A larger quantity of water is preferable for the pulping of the fruits, and is best applied in two or three successive portions. It should be noticed that cassia *pulp* is ordered in B. P., not the *fruit* from which pulp is made. Senna and coriander added last to avoid undue heating.

Questions on Chapter X.

1. Distinguish between evaporation, ebullition, desiccation, distillation, and sublimation.

2. What is the influence of each of the following upon the rate of evaporation?—(*a*) Changes in temperature. (*b*) State of surrounding atmosphere. (*c*) Reduction of pressure. (*d*) Surface of liquid. (*e*) Area of heated surface.

3. Describe the construction of a jacketed steam pan.

4. Of what materials are evaporating pans made? Give the advantages and restrictions upon the use of each.

5. Give a brief description of a vacuum pan.

6. What is "latent heat of steam"?

7. Describe minutely the preparation of Liq. Ferri Perchloridi and Glycerinum Plumbi Subacetatis.

CHAPTER XI

DISTILLATION

When the object of evaporation is to separate and preserve the more volatile constituent, the vapour is passed through a cooling arrangement whereby it is condensed into the liquid or solid state. The operation is then called distillation or sublimation, according as the product is liquid or solid. The essential parts of an apparatus for distillation are (1) the still or retort; (2) the condenser; (3) the receiver.

1. **The Still or Retort.**—This is the part of the apparatus in which the mixture is heated; it is made of glass, earthenware, or metal. Glass retorts are either plain or tubulated, as shown in Fig. 43. The liquid is introduced into the bulb through the tubulure, if tubulated, and the neck fits into the condenser or receiver to be presently described. Ordinary glass flasks are also frequently used, which are then connected with the condenser by means of a bent glass tube passing through a well-fitting cork or india-rubber bung.

Fig. 43.

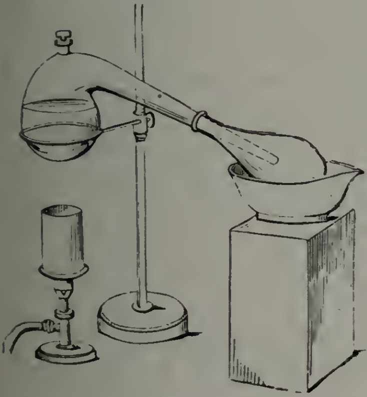
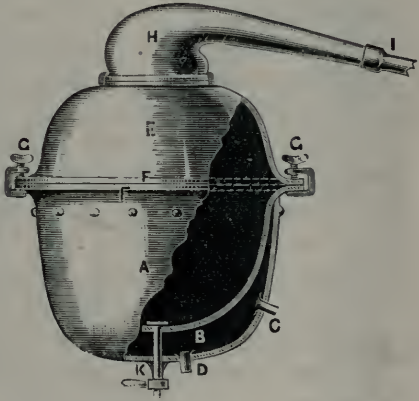


Fig. 44.



Pharmaceutical still.—On account of the danger of fracture, glass retorts are not usually employed for large quantities, their place being supplied by earthenware, or, where applicable, by copper. Lead and platinum stills are also used for particular purposes, such as hydrofluoric acid. Earthenware or stoneware retorts are similar in form to glass ones. Metal retorts, to which the name *still* is usually applied, are almost always constructed of tinned copper; various forms are in use, but the two following will be found most generally useful. Fig. 44 represents an ordinary pharmaceutical still; it may be heated either by the direct heat of a furnace or powerful gas burner, or preferably by being set in a steam jacket, as shown in the figure. A is the body of the still, which is heated by the steam jacket B, steam entering by the jet C, the waste finding an exit at D. E is the cover or *dome*, which is secured by accurately fitting flanges F F, between which a piece of moistened brown paper is placed, and tightly screwed up by the thumb-screws or bolts G, G, of which at least four are needed. Similar flanges secure the *still-head* H, which is bent over to connect with the condenser at I. K is a tap for running out the residue from the distillation. In small stills the dome and head are sometimes made in one piece, or the dome may be a part of the body, but stills so made are not so convenient for cleaning; the flanges, too, are also commonly replaced by making the various parts to fit into each other, the joint being perfected by almond or linseed luting, made by making a stiff "poultice" with boiling water. In this latter case it is necessary to hang weights over the still-head to prevent it from being lifted off by the force from within.

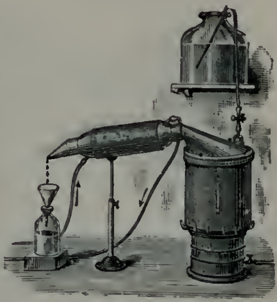
Remington's still possesses some advantages, but it will be more conveniently described after referring to the condenser.

2. **The Condenser.**—The simplest form of condenser is that in which the neck of the retort and the receiver together form the only condenser necessary. Fig. 43 represents such an arrangement. It is only applicable in the case of substances which can be easily condensed without much cooling, *e. g.* nitric acid. The receiving flask is immersed in water or broken ice, and a wet cloth, kept cool by dropping water, may be wrapped around it. Next to this in simplicity is the ordinary Liebig's condenser, which consists of a long glass

tube (Fig. 47), represented by the dotted line, passing through a metal jacket G, through which cold water is continually flowing in the direction from H to I. The ends of the water-jacket are made tight by rubber bands J, J, secured by wires bound tightly around. The retort neck is fitted into the tube, and the joint may be secured by luting, or by thin rubber sheeting tied around. If the neck be too large an *adapter* must be used (K, Fig. 47).

Remington's still.—This form of condenser entails inconvenience from its great length; the *Remington* still, previously alluded to, obviates this difficulty by introducing the principle of the tubular boiler. Fig. 45 represents this still.

FIG. 45.



The body of the still is made of tinned copper, the bottom being flat. A glass tube level on the side of the still shows when the liquid has been distilled to a dangerously low point.

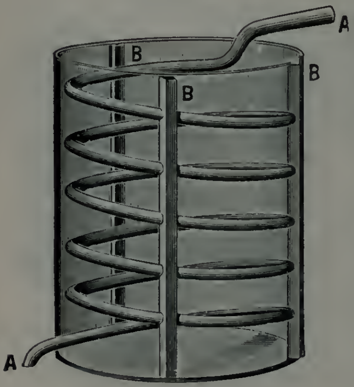
The *still top* differs from most others in having the opening for the escape of vapours drawn over to one side instead of in the centre. By this arrangement the condensing surface of the dome is reduced to a minimum, and condensation *inside the still* is obviated as far as possible.

In the construction of the condenser the single tube of Liebig's is replaced by *seven* parallel ones of small diameter, constructed of block tin, so as to present a greater surface to the cooling action of the condensing water. When a moderate heat *below the boiling-point of water* is required, the body of the still is placed in a water-jacket, and if the quantity of liquid is not large, a round-bottomed copper pan is clamped between the still body and the head, the body of the still being used as a water-bath to heat this pan. An automatic feeding attachment is connected with the tubulure of the still, consisting of a glass syphon, connected by a rubber tube with a glass tube passing into the still, and regulated by a pinch-cock. The bulk of the liquid being contained in a reservoir above the still, the still body is about half filled with liquid, and the heat and cold water turned on until the distillate begins to flow in a steady stream ; the level of the liquid on the gauge is marked, and the pinch-cock so regulated that the inflow of liquid exactly compensates for the loss by distillation. The still may then be left to itself.

For operations where a solid, such as a fresh herb, is present in the still, a wire cage is supplied to contain such substance, and prevent it from touching the bottom of the still and so becoming burnt.

The worm condenser.—Another form of condenser is the *worm*, which is almost universally attached to ordinary metal stills. It is made of block tin or well-tinned copper, and is illustrated in Fig. 46, which represents the worm A A supported in position by the wooden uprights B, B, B, the front of the worm-tub being cut away to show the worm. A constant supply of water circulates through the tub.

Fig. 46.



We have already noted (p. 112) the fact that the condensation of steam (or other vapour) to the liquid state is accompanied by the evolution of a great amount of heat previously latent ; hence the necessity for an abundant supply of cold water around the condenser, the cooling required being exactly equal to the heating previously needed for the production of the vapour.

The Receiver.—*The receiver* has been already several times referred to ; any suitable vessel may be employed. In the case of very volatile liquids the receiver should be immersed in cold water or a mixture of roughly powdered ice and salt, and the end of the condenser tube should always pass well into the neck of the receiver. The liquid which collects in the receiver is called the *distillate*.

Aqua Destillata.—The simplest operation of distillation is the preparation of distilled water. This is usually conducted in a copper still heated by a furnace or by steam under pressure. The first portion which distils should be rejected, as it usually contains considerable traces of ammonia; and the distillation is stopped when about nine tenths of the water has been evaporated, as the latter portions are liable to be contaminated with empyreumatic matter or hydrochloric acid, from the decomposition of organic matter or chlorides of magnesium and calcium. The still used for distilled water should be used for nothing else.

Medicated Waters.—The greater number of the medicated waters are prepared by distilling the drug or its essential oil with water. The student should prepare one or more of these.

Aqua Menthæ Piperitæ.

Take of—

Oil of peppermint	1½ fl. dr.
Water	1½ galls.

Distil one gallon.—B. P.

The distillation may be carried out in an ordinary pharmaceutical still or the Remington still. The distillate will contain oily drops which slowly rise to the surface, but should not be removed by filtration, the water being drawn off from below as required. Especially should filtration with the aid of magnesia or other powders be avoided; magnesia combines with some oils, and all these powders tend to reduce the strength of the waters.

Aqua Cinnamomi.

Take of—

Cinnamon bark	1 part.
Water	16 parts.

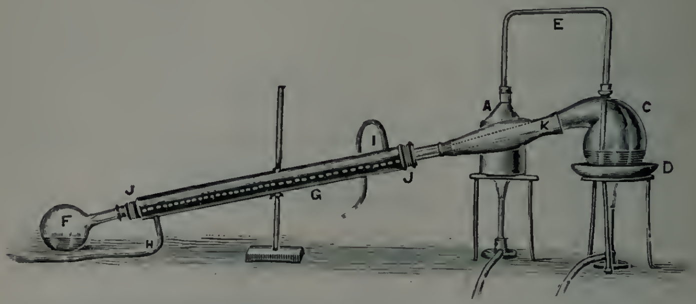
Distil 8 fluid parts.—B. P.

This operation, and all similar ones in which the drug is put into the still, should not be conducted in stills heated by a direct flame or furnace, unless the drug is contained in a close wire cage, a steam-jacketed copper still being the best.

It was formerly customary to allow the drug to soak in the water for a night and a day before distillation; this was called the "*Nychthemerum process.*" It is distinctly advantageous with hard drugs like anise.

Distillation by Injection of Steam.—Another plan is to add only sufficient water to soften the drug, and inject steam into the still, which is also gently heated by a steam jacket. This operation may be imitated by the student who has not access to metal stills in the manner indicated in Fig. 47. A is a tin can containing water for the generation of steam, heated by a Bunsen burner; E is a glass tube passing to bottom of the retort C, which contains the cinnamon and about one-third of the water, gently heated by means of a sand or water bath D. G is the condenser, and F the receiver.

Fig. 47.



In the case of oil of cinnamon the excess of oil falls to the bottom, it being of greater specific gravity than water.

The following is a tabulated statement of the methods of preparation of the Pharmacopœial medicated waters.

Name.	Proportion of water used.	Proportion of water distilled.
Aqua Anethi	20 to 1 of fruit	10 to 1 of drug.
,, Anisi	20 to 1 of fruit	10 to 1 of drug.
,, Aurantii Flor.	Imported, obtained by distillation.	
,, Carui	20 to 1 of fruit	10 to 1.
,, Cinnamomi	16 to 1 of bark	8 to 1.
,, Fœniculi	20 to 1 of fruit	10 to 1.
,, Laurocerasi	3⅛ to 1 of fresh leaves	1¼ to 1. See "Standardised Preparations."
,, Menthæ Piperitæ	1280 to 1 of oil	853 to 1.
,, ,, Viridis	1280 to 1 of oil	853 to 1.
,, Pimentæ	23 to 1 of fruit	11½ to 1.
,, Rosæ	5 to 1 of fresh petals	1 to 1.
,, Sambuci	5 to 1 of fresh flowers	1 to 1.
,, Camphoræ	320 to 1	1000 to 1 (about). Prepared by solution (see page 77).
,, Chloroformi	200 to 1 (measure)	200 to 1 (measure). Prepared by solution (see page 59).

Volatile Oils.—The volatile oils constitute another important class of preparations involving distillation; the following description of the process of distilling oil of rosemary, as adopted by Mr. Sawer, of Brighton, is taken (abridged) from the 'Pharmaceutical Journal' of January 25th, 1890. It affords a good example of the manner of dealing with fresh herbs.

For the purposes of distillation the young shoots are cut at the end of August or beginning of September, and separated from the wood, *i. e.* the ends of the main branches, as much as possible. The twigs are then packed tightly into a perforated copper vessel, which is covered with a perforated copper lid, and the whole is lifted into the still by pulley tackle. If the rosemary be not distilled soon after being gathered it is liable to heat. Cold water is let into the still until it rises nearly to the level or within an inch of the lid; the head of the still is then luted on and clamped, and the mass left to become saturated till next morning. The fire is then lit, and when the water begins to boil the oil distils over. That which comes over during the first twenty-five or thirty minutes is the finest; that which comes over afterwards is small in quantity and inferior in quality. A worm of tin pipe in a galvanised iron cylinder is used as a condenser.

The Pharmacopœial essential oils prepared by this or a similar form of distillation are Ol. Anethi, Anisi, Anthemidis, Cajuputi (imported), Carui, Caryophylli, Cinnamomi, Copaibæ,* Coriandri, Cubebæ, Eucalypti (imported), Juniperi, Lavandulæ, Menthæ Piperitæ, Menthæ Viridis, Myristicæ, Pimentæ, Pini Sylvestris (imported), Rosmarini, Rutæ, Sabinæ, Santali, Sinapis, and Terebinthinæ* (imported).

The student should prepare oil of cloves, caraway, or dill, these being yielded in considerable quantity. Operating upon a pound in a small copper still, he should allow the distillate to stand in a narrow vessel till the oil has collected, having previously dissolved about one-sixth its weight of salt in the distillate, which causes the oil to separate more perfectly. In this way the yield should be about 2 oz.,

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* In these cases the crude oleo-resin being employed, a "cage" cannot be used; the best plan of distillation is by injection of steam.

½ oz., and ¼ oz. respectively. On the large scale it is better not to employ salt, but to use the water to put into the still for the next batch of oil required.

The remaining preparations of the Pharmacopœia in which distillation forms an essential part of the process may be divided into four sections:—1. Those requiring simple distillation similar to that already described, but omitting the water. 2. Distillation accompanied by chemical action in the still. 3. Fractional distillation. 4. Destructive distillation.

The remaining preparations made by simple distillation are—

Æther Purus.—Ether is first washed twice by agitation with half its bulk of water, removing the watery layer after each washing (to remove spirit); it is then introduced into a retort together with recently burned lime and chloride of calcium (which combine with the dissolved water), allowed to stand twenty-four hours, and then distilled from a water-bath by a gentle heat. Sp. gr. ·720.

Alcohol Ethylicum.—1 part of dried carbonate of potassium is added to 10 fl. parts of rectified spirit and agitated frequently during twenty-four hours; this removes a portion of the water forming a heavy layer of liquid beneath the spirit. This spirit is decanted into a flask containing 8 parts of recently fused chloride of calcium, securely corked and allowed to stand twenty-four hours, agitating from time to time; then 1 part is distilled with a *dry* condenser, returning this to the flask, and finally distilling 7½ fl. parts. Sp. gr. ·797 to ·800. Lime is preferable to chloride of calcium for the latter part of this operation.

Spiritus Ammoniæ Aromaticus.—Carbonate of ammonium 4 parts, strong solution of ammonia 8 fl. parts, volatile oil of nutmeg 0·55 fl. part, oil of lemon 0·81 fl. part, rectified spirit 120 fl. parts, water 60 fl. parts. Distil the spirit, oils, and water until 140 fl. parts have passed over, then separately another 9 fl. parts. Dissolve the carbonate of ammonium in this second distillate together with the strong ammonia solution by gently warming it to about 60° C. in a securely stoppered bottle. Filter when cold through cotton wool, and mix it with the 140 parts of former distillate. The whole should measure 160 fl. parts when completed. Sp. gr. ·896.

Spiritus Ammoniæ Fœtidus.—Asafœtida 1½ parts, strong solution of ammonia 2 fl. parts, rectified spirit a sufficiency to produce 20 fl. parts.

Having broken the asafœtida into small pieces, leave it in contact with 15 fl. parts of the spirit in the still for twenty-four hours, then distil off the spirit, add the ammonia to the distillate, and finally sufficient S. V. R. to produce 20 fl. parts. Sp. gr. about ·847.

Spiritus Armoraciæ Compositus.—Fresh horseradish root, scraped, 1 part, bitter orange peel, cut small and bruised, 1 part, nutmeg $\frac{1}{40}$ part, proof spirit 8 fl. parts, water 3 parts. Mix, and distil 8 fl. parts. Sp. gr. about ·920.

2. Those cases of distillation accompanied by chemical action in the still are in some cases more suitable for study under the science of chemistry; these will be simply described by equations.

Nitric acid.—$NaNO_3 + H_2SO_4 = NaHSO_4 + HNO_3$.
$NaHSO_4 + NaNO_3 = Na_2SO_4 + HNO_3$.

Phosphoric acid.—$3P_4 + 20HNO_3 + 8OH_2 = 12H_3PO_4 + 20NO$.

The distillation in this case is used to economise nitric acid, the phosphoric acid remaining in the retort.

Æther.—$C_2H_5OH + H_2SO_4 = C_2H_5HSO_4 + OH_2$.
$C_2H_5HSO_4 + C_2H_5OH = C_2H_5OC_2H_5 + H_2SO_4$.

Æther Aceticus.—$C_2H_5OH + CH_3COONa + H_2SO_4$
$= CH_3COOC_2H_5 + NaHSO_4 + OH_2$.

Chloroform.—$4C_2H_5OH + 8CaCl_2O_2 = 2CHCl_3 + 3(HCOO)_2Ca + 5CaCl_2 + 8OH_2$.

Chloroform is now frequently prepared from acetone.

The four following, although belonging to the same category as the above, may readily be prepared in the pharmaceutical laboratory.

Hydrobromic acid.—Sulphuretted hydrogen, obtained by the action of dilute sulphuric or hydrochloric acid upon sulphide of iron, is passed through a mixture of bromine with fifteen times its volume of water until the red colour has disappeared. The liquid is filtered and then distilled, the first portions being rejected until the odour of SH_2 or SO_2 is absent, then collected until sulphuric acid begins to distil. The distillate is then diluted with water until of sp. gr. 1·077 at 15·5° C. The acid is improved by redistillation before dilution, with a few grains of barium carbonate in the retort to retain traces of sulphuric acid.

$$4SH_2 + 10Br_2 + 8OH_2 = 20HBr + 2H_2SO_4 + S_2.$$

Bromide of phosphorus is formed as an intermediate substance, but is decomposed by excess of SH_2 and OH_2.

Hydrocyanic acid.—*Vide* Standardised Preparations.

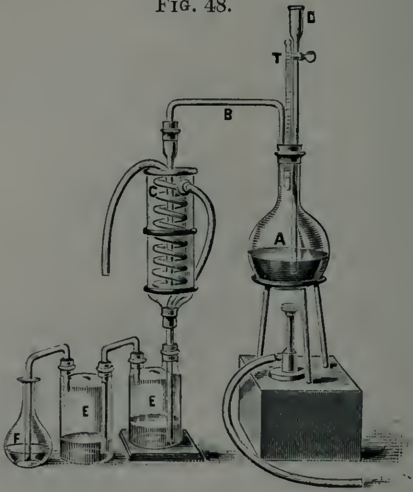
Spiritus Ætheris Compositus.—*Vide* B. P. Consists of ethereal oil and spirit of ether.

Spiritus Ætheris Nitrosi.—Nitric acid 3 fl. parts, sulphuric acid 2 fl. parts, copper in fine wire (about No. 25) 2 parts, rectified spirit a sufficiency. To 20 fl. parts of the spirit add gradually the sulphuric acid, stirring them together; then add in the same way 2½ fl. parts of the nitric acid. Put the mixture into a retort or (preferably) a flask, into which the copper has been introduced, and to which a thermometer is fitted. Attach now an efficient condenser, and applying heat gently, let the spirit distil at a temperature commencing at 170° F. (76·7° C.) and rising to 175° F. (79·4° C.), but not exceeding 180° F. (82·2° C.) until 12 fl. parts have paased over and been collected in a bottle, the latter and the condenser being kept cool with ice-cold water; then withdraw the heat, and having allowed the contents of the retort to cool, introduce the remaining ½ fl. part of nitric acid and resume distillation as before, until the distilled product has been increased to 14 fl.

parts. Mix this with 40 fl. parts of S. V. R., or as much as will make the product correspond to the nitric oxide test.* Preserve it in well-closed vessels.

Prepared as above by the B. P. directions a great loss of nitrous ether occurs, and consequent lessening of the product, on account of the great volatility of nitrite of ethyl. Fig. 48 represents a more economical arrangement, the single receiver being replaced by *three* in succession, so as to ensure the absorption of any ethyl nitrite which escapes condensation in the first. A is the generating flask, fitted with a thermometer T, and a tapped funnel D to introduce latter portion of the nitric acid; a bent tube B connects A with the spiral condenser C, the lower end of which passes into the first receiver E and below the surface of the spirit it contains (20 fl. parts). This receiver connects with another (both known as Woulff's bottles), and this with a third, which may be a bottle or flask loosely plugged with cotton wool, these two containing each 6 fl. parts of spirit. (See Farr, 'P. J.' [3], xix, p. 440, Dec. 1st, 1888.) Ethyl nitrite is not the only product, the reaction being complicated by secondary actions; aldehyde is an important constituent of the finished preparation. Spirit of nitrous ether should be kept in well-closed bottles in a dark place.

FIG. 48.



3. **Fractional Distillation.**—It is frequently necessary in distillation to separate the portions distilling at different temperatures; this operation is called *fractional distillation,* and is readily carried out by using a properly constructed flask or "fractionating" tube, a thermometer being attached, the bulb of which reaches to just below the side tube connecting with the condenser. Fig. 49 represents two forms of frac-

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* For the estimation *vide* Standardised Preparations.

tionating tubes, and Fig. 51 a fractionating flask. Fig. 50 shows a form of fractionating tube (Glynsky's), each bulb of which is supplied with a glass bead, which forms a valve by resting upon the constricted portion below.

Fig. 49.

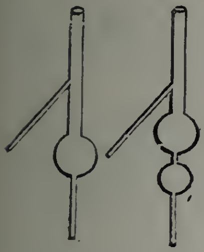


Fig. 50.

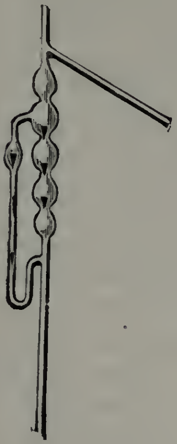
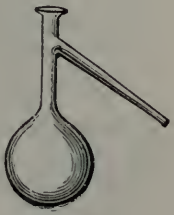


Fig. 51.



On the large scale it is usual to pass the vapour issuing from the retorts through a worm arranged above, surrounded by a bath of the vapour of some liquid boiling at the desired temperature. In this way products of higher boiling-point are condensed and returned to the still; this is called "fractional distillation with still-head of constant temperature."

There are a few Pharmacopœial articles prepared by fractional distillation besides hydrobromic acid and spirit of nitrous ether, which are imperfect examples. Amylic alcohol and nitrite of amyl are cases in point. Carbolic acid and butyl chloral are also so prepared, but are products of the manufacturing chemical laboratory, unsuited for treatment in this work.

Amylic alcohol.—Fusel oil from the distilleries is first distilled at 100° C., the residue washed with brine to remove propylic alcohol, which is soluble; the upper layer is removed and introduced into a fractionating flask. Distillation is proceeded with until the thermometer registers 128° C., when the receiver is changed, and the portion distilling between this and 132° C. alone reserved as amylic alcohol, the flask and fractionating tube being carefully protected from draughts. Sp. gr. 818 (nearly).

Amyl Nitris.—Amylic alcohol, obtained as above, is best converted into the nitrite as follows. $6\frac{1}{8}$ parts by weight of sulphuric acid are gradually added to 11 parts by weight of amylic alcohol, keeping cool by immersion in water. When quite cold this mixture is poured slowly, with constant stirring, to the bottom of a solution of 9 parts of sodium nitrite dissolved in 27 parts of water, by means of a thistle-headed tube, keeping the whole cool by immersion in ice-cold water. Amyl nitrite rises to the surface, and may be removed by a pipette or syphon. It is washed with a solution of potassium carbonate (to remove acid), then dried by standing over dry carbonate of potassium, and finally distilled from a fractionating flask. That which distils between 90° C. and 100° C. is collected, preserved in well-stoppered bottles, kept upside down in a cool place.

$$C_5H_{11}OH + H_2SO_4 = C_5H_{11}HSO_4 + OH_2.$$

$$C_5H_{11}HSO_4 + NaNO_2 = C_5H_{11}NO_2 + NaHSO_4.$$

See also papers by Dunstan and others, 'Pharm. Journ.' (3), xix, p. 845, et seq., December 22nd, 1888; and Williams and Smith, 'Pharm. Journ.' (3), xvi, p. 499, December 12th, 1885.

4. **Destructive Distillation.**—This is distillation whereby compound substances, usually organic, are heated to a temperature at which decomposition occurs, resulting in the production of distillates which did not exist ready formed in the original substance. Soft paraffin from petroleum, hard paraffin from shale, acetic acid and creasote from wood, *huile de cade* from wood of *Juniperus oxycedrus*, and liquid pitch from wood of species of pines, are examples from the B. P.; others are coal gas, coal tar, methyl alcohol, &c. It is usually carried out in cast-iron retorts strongly heated by furnaces.

Questions on Chapter XI.

1. Describe the construction of a metal still with condensing worm.
2. How does the "Remington" still differ from those commonly employed?
3. Describe the preparation of Aqua Anethi; name the other waters of the B. P. prepared similarly.
4. How does cherry laurel water differ from the other waters of the B. P.?
5. How would you prepare oil of pimento?
6. Describe the preparation of oil of copaiba.
7. Describe the preparation of Spt. Ammon. Aromat.
8. Write equations for the production of phosphoric acid, hydrobromic acid, and acetic ether.
9. What is understood by fractional distillation and destructive distillation?
10. Describe the preparation of spirit of nitrous ether.
11. What is the object of the following?—Chloride of calcium and lime in preparation of pure ether, carbonate of potassium for ethylic alcohol, copper in spirit of nitrous ether.

CHAPTER XII

SUBLIMATION—EXSICCATION

I. *Sublimation.*

Sublimation.—Closely allied to distillation is *sublimation,* the difference being that in sublimation the product obtained is a *solid* instead of a *liquid,* and therefore the still and condenser must be replaced by more suitable apparatus, or the worm would soon be blocked by the *sublimate.* The apparatus consists of two parts, a heating pot or retort, and a cooling surface. If the object is to obtain the sublimate in a mass, as with perchloride of mercury, the cooling surface is made small, so as to condense the vapour at a temperature not much below its congealing point; if, however, fine crystals are desired, as little heat as possible is applied, and the condensation allowed to take place slowly; lastly, if desired in fine powder, the vapour is conducted into a large chamber where it can be rapidly cooled by draughts or other artificial means, *e. g.* sulphur.

The following B. P. articles are prepared by sublimation:

Name of preparation.	Source.	Equation.	Form for use.	Remarks.
Acidum Arseniosum	Arsenical ores	$2FeSAs + 3O = 2FeS + As_2O_3$	Fine powder or lumps	In current of air.
Acidum Benzoicum	Gum benzoin	—	Crystals	Exists in benzoin in free state.
Ammonii Carbonas	Gas liquor, hydrochloric acid, and chalk	$2CaCO_3 + 4NH_4Cl = 2CaCl_2 + NH_3 + NH_4HCO_3, NH_2,COONH_4 + OH_2$	Crystalline cakes	—
Ammonii Chloridum (purification)	Gas liquor	$NH_4HO + HCl = NH_4Cl + OH_2$	Crystalline cakes	—
Camphor (purification)	Crude camphor	—	Masses	—

Name of preparation.	Source.	Equation.	Form for use.	Remarks.
Hydrargyri Perchloridum	Mercuric sulphate 5, sodium chloride 4, manganese dioxide ¼	$HgSO_4 + 2NaCl = HgCl_2 + Na_2SO_4$	Crystalline masses	—
Hydrargyri Subchloridum	Mercuric sulphate 10, mercury 7, sodium chloride 5	$Hg_2SO_4 + 2NaCl = 2HgCl + Na_2SO_4$	Fine powder	—
Iodum (purification)	Crude iodine	—	Crystals	—
Sulphur Sublimatum	Crude sulphur or pyrites	$2FeS_2 = 2FeS + S_2$	Fine powder	—

The preparation of most of these is fully described in works on chemistry; the student of pharmacy should, however, prepare at least two of these, as the experience is valuable.

Acidum Benzoicum.—The sublimation of benzoic acid may be imitated on the small scale by mixing one part of gum benzoin with about two parts of sand (to prevent burning), and introducing the mixture into a shallow tin resting on a sand-bath. The tin is covered with a perforated paper, and over this is placed a cone of stout paper, or a funnel with the end closed. Heat is cautiously applied to the sand-bath, when the benzoic acid passes off in vapour and condenses in feathery crystals upon the cone or funnel. When cold it may be removed.

Hydrargyrum Perchloridum.—This may be experimentally prepared by mixing 20 parts of persulphate of mercury, 16 parts of salt, and 1 of black oxide of manganese, introducing them into a gallipot or "honeypot," covering with a similar pot, and securing the joint from leakage by luting made of moistened fire-clay. The luting is slowly dried, and the lower pot set in a sand-bath, the sand being heaped up to the *top* of the luting, then heated by a good gas flame for an hour or two. An ordinary Bunsen flame is not sufficient, one of Fletcher's radial gas burners or the burner attached to his "water-heater" being better. The black oxide of manganese is added to prevent the formation of any subchloride of mercury.

Iodum.—The crude iodine is purified by mixing with a small quantity of iodide of potassium and subliming in a similar manner to perchloride of mercury, but using far less heat. The iodide of potassium serves to retain any chlorine or bromine which exists in the crude iodine, thus: $2KI + Cl_2 = 2KCl + I_2$.

Camphor.—Crude camphor is obtained by heating the camphor wood with steam, when the camphor and "camphor oil" distil with the steam. The oil and water are drained off from the camphor, which is re-sublimed for use in pharmacy. The "camphor oil" yields a further supply of camphor if cooled to a low temperature.

II. *Exsiccation.*

Exsiccation or Desiccation.—*Desiccation* or *exsiccation* is the removal of water from solids; the term is rightly applied only in those cases in which the water is mechanically adherent, or exists as water of crystallisation (see Chap. XV). It is almost always accomplished by heating in a steam or water bath, or in an iron pan or sand-bath over a slow fire.

Calcination.—*Calcination* is the operation of removing chemically combined water or other volatile constituents such as carbon dioxide, from solids by the application of a strong heat; if applied together with a current of air this operation is frequently called *roasting,* and is a common preliminary in the treatment of ores. The term calcination is only used when the substance does not melt.

Official Preparations.—The following table contains the official preparations made by these processes combined with methods already noticed; the student should prepare each of those marked *.

Name of preparation.	Temperature.	Reaction.	Product.	Remarks.
Alumen Exsiccatum	Below 400° F. (204·4° C.)	$Al_2(SO_4)_3K_2SO_4,24OH_2 = Al_2(SO_4)_3K_2SO_4 + 24OH_2$	54 to 55 per cent.	Note 1.
Ammonii Nitras	Below 320° F. (160° C.)	$NH_4NO_3,OH_2 = NH_4NO_3 + OH_2$	—	—
*Calamina Præparata	Dull red heat	Organic matter $= CO_2, OH_2$, &c.	Variable	Note 2.
Calcii Sulphas	Dull redness	$CaSO_4,2OH_2 = CaSO + 2OH_2$	Nearly 80 per cent.	—
Calx	White heat	$CaCO_3 = CaO + CO_2$	About 56 per cent.	Note 3.
Calx Sulphurata	Bright redness	$CaSO_4 + C_2 = CaS + 2CO_2$ (incomplete)	—	Notes 4 and 6.
Carbo Animalis	Redness	Organic matter $= C + CO_2 + OH_2$, &c.	—	Note 6.
Carbo Animalis Purificatus	Redness	$Ca_32PO_4 + 4HCl = 2CaCl_2 + CaH_4(PO_4)_2$	About 10 per cent.	Notes 5 and 6.
Carbo Ligni	Redness	Organic matter $= C + CO_2 + OH_2$, &c.	—	Note 6.
*Ferri Sulphas Exsiccata	212° F. (100° C.)	$FeSO_4,7OH_2 = FeSO_4,OH_2 + 6OH_2$	About 62 per cent.	Note 7.
Hydrargyri Oxidum Rubrum	About 600° F. (315° C.)	$Hg(NO_3)_2 + Hg = 2HgO + 2NO_2$	—	Note 8.
Hydrargyri Persulphas	About 620° F. 327° C.)	$Hg + 2H_2SO_4 = HgSO_4 + SO_2 + 2OH_2$	—	—

Name of preparation.	Temperature.	Reaction.	Product.	Remarks.
*Liquor Sodii Arseniatis	Below 300° F. (148·9° C.)	$Na_2HAsO_4,12OH_2 = Na_2HAsO_4 + 12OH_2$, or $Na_2HAsO_4,7OH_2 = Na_2HAsO_4 + 7OH_2$	46·25 per cent. 59·6 per cent.	Note 9.
Magnesia Levis	Low redness	$(MgCO_3)_3Mg(HO)_2,4OH_2 = 4MgO + 3CO_2 + 5OH_2$	42 per cent.	Note 3.
Magnesia Ponderosa	Low redness	Ditto	42 per cent.	Note 3.
Os Ustum	Bright redness	Organic matter + O = $CO_2 + OH_2$, &c.	—	Note 3.
Sodii Carbonas Exsiccata	Sand-bath	$Na_2CO_3,10OH_2 = Na_2CO_3 + 10OH_2$	37 per cent.	Note 10.
*Zinci Oxidum	Low redness	$ZnCO_3(Zn2HO)_2H_2O = 3ZnO + CO_2 + 3H_2O$	73 per cent.	Note 3.

Note 1.—If the temperature be allowed to exceed 204·4° C. oxysulphate or even oxide of aluminium may be produced, with consequent insolubility of the product. Ammonium alum cannot be used, as the ammonia is volatilised.

Note 2.—The ore is first separated as far as possible from quartz (the nearly colourless crystalline portion), then calcined in a covered crucible, powdered, and freed from coarser particles by elutriation (see p. 43). If an open crucible were used, much of the carbonate would be converted into oxide of zinc. The product should dissolve almost entirely in dilute HCl.

Note 3.—These must be calcined in a furnace so arranged that a draught may be constantly passing over or through the substance, so as to carry off the carbon dioxide formed. If it be attempted in a closed crucible the first portions of CO_2 evolved form an atmosphere in which even an intense heat scarcely effects the further decomposition of the carbonates; this is particularly marked in the case of lime. The operation may be imitated on the small scale by using a crucible perforated so as to allow the draught to pass upwards through the contents.

Crucibles.—*Crucibles* (from *crux*, a cross, or an instrument of torture; probably referring to the high temperatures employed during their use) are made of specially prepared earthenware, porcelain, black-lead, or platinum, the first being commonly employed for large quantities. One form of crucible is shown within the furnace in Fig. 52.

Gas Furnace for Calcination.—A useful furnace for such operations with or without a crucible is Fletcher's reverberatory furnace, which is shown in Fig. 52, with one of the doors removed to show contents. The burner is at one end, out of the way of injury in case of accident to a crucible. Crucibles, &c., stand on the solid bottom of the furnace, perfectly steady and firm. It may be used with or without a blast of air from a foot or other form of bellows.

Note 4.—The heat must be continued until the black colour has entirely disappeared. Sugar may be advantageously used in place of the charcoal.

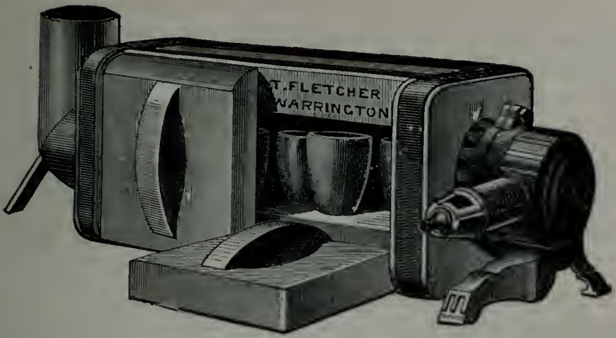


Fig. 52.—Reverberatory furnace, with burner, and containing crucibles.

Note 5.—These operations must all be conducted in a covered crucible, or much loss will occur, the carbon being oxidised by the air.

Note 6.—The phosphate and carbonate of calcium are mostly removed from animal charcoal by repeated digestion in dilute HCl, the carbon washed, dried, and ignited. It is a practical impossibility to obtain an article answering the B. P. test, "about 2 per cent. of ash."

Note 7.—At a higher temperature sulphate of iron loses the remaining molecule of water, partly in combination with the sulphuric radical.

Note 8.—Mercury, 4 parts, are dissolved in nitric acid 4½ fl. parts, and 2 parts of water; evaporated to dryness, uniformly mixed with 4 parts more mercury, and then gently heated in a porcelain dish till nitrous vapours no longer escape. A little should be tested by heating in a dry test-tube; no orange vapours should appear.

Note 9.—The desiccation should be slowly conducted, raising the temperature very gradually at first, and carefully avoiding any excess of heat, or pyroarseniate may be formed, thus: $2Na_2HAsO_4 = Na_4As_2O_7 + H_2O$, with consequent increase in strength. Desiccation should be continued until two successive weighings of the dish and its contents at quarter-hour intervals are identical. *When dried* dissolve 1 part of the salt in sufficient distilled water to produce 100 fl. parts = 1 in 100.

Note 10.—Should be first exposed to very gentle heat, as for arseniate, so as to avoid melting the crystals (not over 24° C.), and when they crumble increase gradually till perfectly dry (rather over 200° C.).

The operation of desiccation or drying forms an important part of the processes of manufacture of a large number of preparations which are more correctly classified under different headings; such, for example, as the granular effervescent compounds, scale preparations, precipitated compounds like phosphate of iron, and the drying of fresh drugs already considered. There is a class of preparations whose successful production depends very largely on the operation of drying, which will now be noticed, viz. the lozenges.

Lozenges.—The manufacture of lozenges is now practically entirely in the hands of the manufacturing confectioner, but the pharmacist is occasionally required to prepare them from special recipes. The method of operating is illustrated by the following two examples from the B. P., which should be practically carried out by the student:

"*Trochisci Acidi Benzoici.*

"Take of—

Benzoic acid	360 grs.
Refined sugar in powder	25 oz.
Gum acacia in powder	1 oz.
Mucilage of gum acacia	2 fl. oz.
Distilled water	a sufficiency.

"Mix the benzoic acid, sugar, and gum, add the mucilage and water to form a proper mass. Divide into 720 lozenges, and dry them in a hot air chamber at a moderate temperature."

When a very active substance forms one of the ingredients it is first dissolved in water, if soluble, and the solution mixed with the other ingredients, as in the following:

"*Trochisci Morphinæ.*

"Take of—

Hydrochlorate of morphine	20 grs.
Tincture of tolu	½ fl. oz.
Refined sugar in powder	24 oz.
Gum acacia in powder	1 oz.
Mucilage of gum acacia	a sufficiency.
Distilled water	½ fl. oz.

"Dissolve the hydrochlorate of morphine in the water; add this solution to the tincture of tolu, *previously* mixed with two fluid ounces of the mucilage (gradually added); then add the gum and sugar, previously mixed, and more mucilage, if necessary, to form a proper mass. Divide into 720 lozenges, and dry these in a hot air chamber at a moderate temperature." The water must be warm.

Several points in the above process require comment. The massing may be done in a mortar having a long pestle, in the same way that pill masses are kneaded, or in one of the many kneading machines which are supplied by makers of pharmaceutical appliances. The division may be accomplished as follows :—The mass is pressed, rolled, or beaten out on a slab into a flat cake, and made of as nearly uniform thickness as possible, by means of a cylindrical "rolling-pin," having ridges at each end to prevent the cake from being rolled too thin; or the *slab* may have similar ridges. A little of the mixed sugar and gum is sprinkled over the surface to prevent adhesion to the cutter, which consists of a nearly cylindrical metallic cone, the narrower end of which is sharpened. Several of these cones may be kept, of various sizes, so as to meet the call for lozenges of different weights. By pressing this cone into the flattened mass, circular discs will be cut, which can be removed by inverting the cone after several have been made. Oval or oblong "tubes" may also be used, if preferred.

The drying may be effected at first by simple exposure on wire trays to a dry atmosphere, and then in the drying closet previously described (p. 24), the temperature being very slowly raised and regulated not to exceed 50° C.

Lozenges can, of course, be much more accurately made by suitable machinery, but the above process answers the requirements of the retail pharmacist in most cases.

Official Lozenges.—The following are the lozenges of the British Pharmacopœia, with strength :

Troch.	Morphinæ	$\frac{1}{36}$	grain	in each.
,,	,, et Ipecacuanhæ	$\left\{\begin{matrix}\frac{1}{36}\\ \frac{1}{12}\end{matrix}\right\}$	,,	,,
,,	Opii	$\frac{1}{10}$	,,	,,
,,	Ipecacuanhæ	$\frac{1}{4}$	,,	,,
,,	Acidi Benzoici	$\frac{1}{2}$	,,	,,
,,	,, Tannici	$\frac{1}{2}$	,,	,,
,,	Catechu	1	,,	,,
,,	Ferri Redacti	1	,,	,,
,,	Santonini	1	,,	,,
,,	Bismuthi (Subnitrate)	2	grains	,,
,,	Potassii Chloratis	5	,,	,,
,,	Sodii Bicarbonatis	5	,,	,,
,,	Sulphuris { Precipitated sulphur	5	,,	,,
	Bitartrate of potassium	1	grain	,,

Questions on Chapter XII.

1. Define sublimation, exsiccation, calcination.

2. Which of the official sublimates are obtained in fine powder? Describe the preparation of each.

3. Explain the addition of iodide of potassium and of manganese dioxide in the preparation of iodine and perchloride of mercury respectively.

4. What is the temperature at which each of the following is prepared:—(1) Ferri Sulphas Exsiccata, (2) Alumen Exsiccatum, and (3) Sodii Arsenias (for liquor)? Give reasons in each case.

5. Describe the preparation of sulphurated lime and red oxide of mercury.

6. Describe the preparation of bismuth lozenges.

7. Give the strength of the official lozenges of (1) opium, (2) santonine, (3) morphine and ipecacuanha, (4) benzoic acid, (5) sulphur.

CHAPTER XIII

FUSION

Melting-point.—When solid substances are heated, there is usually a certain definite temperature at which they become liquid; this is called the "*melting*" or "*fusing* point," and the act of liquefying is called "*fusion*." If capable of fusion a substance is said to be *fusible;* if not, *infusible.*

A great number of pharmaceutical preparations depend upon the operation of fusion for their successful production; these include all the suppositories and most of the plasters and ointments, as well as many chemicals and some unclassified galenicals.

Liquefaction.—In many of these cases—as, for example, the melting of fats and resins for preparing ointments—the truly liquid state is preceded by a pasty or glutinous condition. When this occurs the operation is sometimes specially distinguished by the term "*liquefaction*." This term, in the opinion of the author, is inapt, particularly as some metals, *e. g.* iron, behave similarly.

The operation of fusion is carried out in various kinds of apparatus, according to the temperature needed and other conditions, a dish over a water or steam bath being employed for fats and similar bodies; crucibles are used for metals, and covered iron or enamelled pots, heated by a gas or coal furnace, for some chemicals.

Latent Heat of Fusion.—It has already been stated that heat is rendered latent when water or other liquids are converted into vapour; the same occurs when solids are fused, but not to so great a degree. As an example the student may take a quantity of broken ice, and having placed it in a dish, apply the heat of a water-bath, keeping a thermometer immersed in the mass. The temperature will rise to 32° F. (0° C.) if previously below, after which no appreciable rise occurs in the water which is produced until the whole of the ice has melted. The amount absorbed is in this case called *the latent heat of water*, or, generally, "*latent heat of fusion.*" An exactly similar amount of heat is evolved when a liquid solidifies. The latent heat of water amounts to rather over 79° C., or 142° F.; that is, it requires as much heat to melt a given weight of ice as is needed to raise that quantity of water through 79° C. or 142° F.

Suppositories.—*Suppositories* are conical bodies designed for administration *per rectum*; the medicament is intimately incorporated with an inert substance called the *basis* or *base.* Suppositories are usually prepared with one of the three following bases:—oil of theobroma; soap, starch, and glycerine; or gelatine, water, and glycerine. When applicable, oil of theobroma is preferable on account of its firmness when cold, and ready liquefaction at the temperature of the body. The proportions of the gelatine bases vary with the nature of the drug exhibited; usually suppositories made with it are rather soft, but they melt readily. The soap basis is not to be recommended; it does not melt at the body temperature.

Suppositories are usually prepared at the dispensing counter. The following are the general methods adopted.

1. In preparing suppositories having a basis of soap, glycerine, and starch, first mix the active ingredient with the soap and glycerine of starch, then add powdered starch (preferably that of rice) to produce a suitable mass; unless intended for immediate use the mass should be rather soft, as it hardens by keeping. It is then made into suitable form by hand.

2. When oil of theobroma is the basis, rub about one third of the melted oil with the medicament in a warm mortar, and add this to the remainder of the oil previously melted over a water-bath, preferably contained in a porcelain dish with handle. Stir constantly while cooling, until the mixture is slightly "pasty," and then pour it into the mould, kept cool by ice or other suitable means. The mould consists of metal (usually gun-metal), and is suffi-

ciently explained by the accompanying illustrations, Figs. 53 and 54.

FIG. 53.

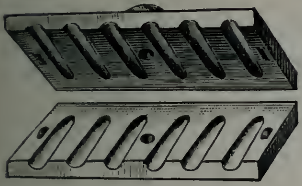
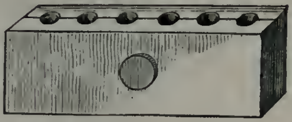


FIG. 54.



The moulds should be previously lubricated by filling with soap liniment or a solution of castor oil in spirit (1 in 5), and then turning the mould upside down to drain for a minute or two. When quite cold unscrew the mould, when the suppositories should be removed without difficulty. The important points in this process are (1) to pour out the mass at the proper temperature, so that the active ingredient shall not sink to the bottom of the mould; and (2) to ensure rapid cooling by ice or a freezing mixture.

3. There is only one instance of gelatine and glycerine as a basis in the B. P., and in this case glycerine itself is the active principle. These are also prepared in a mould, but much larger than those of oil of theobroma, the latter being mostly 15 grains.

The gelatine ½ oz. is placed in a weighed evaporating dish and covered with water; after soaking for two minutes the excess of water is poured off, the gelatine allowed to stand till quite soft, then the glycerine, 2½ oz. is added, and the whole heated over a water-bath till the gelatine is dissolved, and the weight reduced by evaporation to 1560 grains; it is then poured into moulds of the capacity required.

Glycerine suppositories, B. P., are rather soft, and therefore not easy to administer. A firmer article is obtained by dissolving Castile soap, 3 parts, in glycerine, 7 parts, by the aid of heat, and moulding as usual.

The student should prepare at least one example of each of these three classes of suppositories.

Official Suppositories.—The following are the B. P. suppositories:

Name.	Basis.	Strength.	Weight.
Suppositoria Acidi Carbolici c̄ Sapone	Curd soap 15, glycerine of starch q. s.	1 gr.	Nearly 20 grs.
,, Acidi Tannici c̄ Sapone	Curd soap 10, glycerine of starch 3, starch powder q. s.	3 grs.	About 18 grs.
,, Morphinæ c̄ Sapone		½ gr.	About 18 grs.
,, Acidi Tannici .	Oil of theobroma	3 grs.	15 grs.
,, Hydrargyri .		5 grs. mercurial ointment	
,, Iodoformi .		3 grs.	
,, Morphinæ .		½ gr.	
,, Plumbi Composita		3 grs. lead acetate, 1 gr. opium	
,, Glycerini .	Gelatine and water	21 to 84 grs., &c.	30 to 120 grs., &c.

Suppositories containing vegetable extracts may often be incorporated with the gelatine basis, using less glycerine, or with the oil of theobroma if first rubbed smooth with glycerine and water, glycerine and spirit, or spirit and water, before adding the melted fat. See also 'P. J.' (3), xix, pp. 960, 969 (May 25th and June 1st, 1889).

Pessaries.—*Pessaries* (see Chap. XXV) are usually made to weigh 2 drs., and are moulded in a similar manner to suppositories, the basis being commonly oil of theobroma or glycerine and gelatine.

Bougies.—*Bougies* (see Chap. XXV) are similarly prepared, but from their greater length even more care must be taken to pour the compound into the mould at the proper temperature.

Extemporaneous moulds for suppositories, pessaries, and bougies can be made by wrapping tin-foil closely around a "shape" made of wood or wax, setting the tin-foil moulds upright in a bed of sand or other suitable material, and then removing the "shape." Bougies can also be made by piping on a pill machine and rounding off the point.

Before proceeding to notice the plasters and ointments it will be well to introduce here the fatty and waxy substances used in their preparation. In addition to the fixed oils already noticed, these are—

Adeps Præparatus.—"Take of the internal fat of the abdomen of the hog, perfectly fresh, any quantity. Remove as much of the external membranes as possible, and suspend the fat so that it shall be freely exposed to the air for some hours; then cut it into small pieces and beat these in a mortar until they are thus, or by some equivalent process, reduced to a uniform mass, in which the membranous vesicles are completely broken. Put the mass thus produced into a vessel surrounded by warm water, and apply a temperature not exceeding 130° F. (54·4° C.) until the fat has melted and separated from the membranous matter. Finally strain the melted fat through flannel."—B. P.

The exposure to air effects the destruction of an odour which is always present in the freshly slaughtered carcase; the subsequent filtration removes membranous particles, and must be very perfectly carried out to ensure a good article.

Adeps Benzoatus.—Prepared lard 60 parts. Benzoin in coarse powder 1 part. Melt the lard on water-bath, add the benzoin, and, frequently stirring together, continue the application of heat for two hours; finally strain out the residual benzoin by *flannel.* The benzoin is used as a preservative. It has been stated that a good benzoated lard can be made by simply dissolving benzoic acid in lard, when straining is unnecessary; the aroma of such a preparation is not to be compared with that of a well-made B. P. article.

Adeps Lanæ.—Obtained from wool by washing to remove the "yolk," then extracting the wool fat by bisulphide of carbon or other suitable solvent. The crude fat so obtained is purified by filtration through animal charcoal.

Adeps Lanæ Hydrosus.—Wool fat 7 parts, distilled water 3 parts. The water is gradually stirred into the melted wool-fat contained in a warm mortar.

Cera Flava.—Beeswax as it comes from the bee-keepers requires melting by a steam or water bath and straining through fine muslin, or, if much finely-divided impurity be present, through flannel. White wax consists of "yellow wax bleached by exposure to moisture, air, and light."—B. P.

Cetaceum (spermaceti) is obtained from the crude oil from the head of the sperm whale, by cooling, filtering, and pressure, and purified by hot filtration through animal charcoal.

Paraffinum Durum is obtained by distillation of shale and subsequent purification of the last portions of distillate, and *soft paraffin* from petroleum and *oil of theobroma* are also clarified by melting and filtration.

Sevum Præparatum (suet).—This is the internal fat of the abdomen of the sheep purified by melting and straining.

The flannel strainers used in these cases are of the same form as the calico strainer shown in Fig. 31; they are made of thick flannel, with the seams double. When employed for honey or watery fluids the bag should first be wrung out in hot water, but in the case of oils and fats this is of course inadmissible. When tedious filtration is to be effected it is best to suspend the bag in a barrel or box, so as to avoid the drying action of currents of air, whereby the outside frequently becomes covered with scaly or crystalline crusts of dried-up material.

For solid fats or such thick liquids as clarified honey, which is prepared by melting honey on a water-bath and straining, the box containing the strainer should be put in a room or drying cupboard sufficiently warm to retain the substance in the liquid state. This operation of fusion with subsequent straining to remove impurities is sometimes called *depuration* or *despumation*.

Plasters.—*Emplastra* (plasters) consist of the active substance, and the *basis* or adhesive mixture, usually containing oils, fats, or resins, spread upon linen, leather, or some other suitable material, and designed with the object of obtaining a mild but long-continued action of the medicament.

In the preparation of the plasters the general rule is to melt by a water-bath the fatty substances and wax, or the lead, resin, or soap plasters which enter into their composition; to these add the resin and soap previously liquefied by the heat of a sand-bath or small Bunsen flame; finally any powders or other active ingredients, and incorporate thoroughly by stirring whilst cooling.

These last should be added when the melted mass is as cool as will permit of satisfactory admixture.

When water or other aqueous or spirituous liquid enters into the formula, it is generally used as a solvent, and should be removed by subsequent evaporation. Plaster masses usually become harder and more brittle by keeping, especially at the parts exposed to the air; they are therefore commonly made into small rolls, and preferably enclosed in oiled calico. For the same reasons the practice of keeping ready-spread plasters is not to be recommended.

The following table includes the B. P. plaster-masses; those marked * should be prepared by the student.

Name.	Strength.	Basis.	Note.
Emplastrum Picis . .	1 of Burgundy pitch in 2 (nearly)	Frankincense ½, resin, yellow wax, of each $\frac{1}{6}$ (nearly), oil of nutmeg $\frac{1}{26}$, olive oil, water, of each $\frac{1}{13}$ fl. part	
,, Cantharidis .	1 of cantharides in 3	Yellow wax, suet, of each 2½, lard 2, and resin 1	
* ,, Plumbi . .	1 of oxide of lead in 3 (nearly)	Olive oil 2, water 1	1
* ,, Hydrargyri .	1 of mercury in 3 (nearly)	Lead plaster 2, with sublimed sulphur $\frac{1}{164}$, and olive oil $\frac{1}{24}$ (about)	2
,, Ammoniaci c̄ Hydrargyro	1 of mercury in 5 (nearly)	Ammoniacum 4, with sublimed sulphur $\frac{1}{164}$, and olive oil $\frac{1}{24}$ (about)	2
* ,, Belladonnæ .	1 of alcholic extract in 5	Resin plaster and soap plaster, of each 2 parts	3
,, Menthol . .	1 of menthol in 5	Resin 3½ parts, yellow wax ½ part	3
,, Saponis fuscum	1 of curd soap in 6¼ (nearly); 1 of acetate of lead in 3 (nearly)	Olive oil 2 fl. parts, yellow wax 1¼ part	4
,, Saponis . .	1 of curd soap in 7 (nearly)	Lead plaster 6, resin $\frac{1}{6}$	
,, Resinæ . .	1 of resin in 9½	Lead plaster 8, curd soap ½	
,, Opii . .	1 of powdered opium in 10	Resin plaster	3
,, Plumbi Iodidi .	1 of iodide of lead in 10	Lead plaster 8, resin 1	
,, Ferri . .	1 of peroxide of iron in 11	Lead plaster 8, Burgundy pitch 2	
,, Galbani . .	1 each of galbanum and ammoniacum in 11	Lead plaster 8, yellow wax 1	
,, Calefaciens .	1 of cantharides in 26 (nearly)	Resin plaster 13, soap plaster 8, yellow wax, resin, oil of nutmeg, of each 1, water 5	

Note 1.—The ingredients are boiled together for several hours (adding more water if necessary) until the oil and oxide of lead have thoroughly combined, and no pinkish tint can be distinguished. The plaster is then allowed to cool, separated from the water as much as possible, and dried to remove remainder of water. The plaster is chiefly oleate of lead, glycerin being set free, and dissolving in the water. $2(C_3H_53C_{18}H_{33}O_2) + 3PbO + 3H_2O = 3Pb(C_{18}H_{33}O_2)_2 + 2(C_3H_53HO)$.

Note 2.—The sulphur and olive oil are united by heat, then the mercury added and "killed" by trituration till no globules are visible; finally adding the melted ammoniacum or lead plaster.

Note 3.—The melted basis must be as cool as possible before adding the extract or menthol, in one case because of injury to alkaloids, in the other to avoid loss by volatilisation.

Note 4.—The acetate of lead is produced by boiling oxide of lead, 1½ parts, with vinegar, 16 parts, and evaporating with the soap till nearly all the moisture is dissipated; then add other ingredients and continue evaporation until of plaster consistence. $PbO + 2CH_3COOH = (CH_3COO)_2Pb + OH_2$.

The spreading of plasters comes rather within the domain of dispensing, but a work on pharmacy would not be complete without some notice of the method. The first point of consideration is the medium on which it is to be spread; if not otherwise ordered, sheepskin (commonly known as plaster leather) is employed, except in the case of blisters, which are spread upon ordinary adhesive plaster (itself spread on glazed calico); chamois leather, swansdown, brown paper, calico, linen, &c., are occasionally used.

The leather must be selected with as even a surface as possible. A pattern of brown paper enclosing a space of the size and shape required is now cut, and damped by immersion in water for a moment, then laid on the leather and carefully pressed down evenly all round; the corners are preferably cut round. It is now necessary to spread the plaster, which is done by means of a special spatula. Two kinds of spatulas are in use; the older kind (Fig. 55) is heated by a gas stove or other suitable means, while the newer is self-heating; being hollow, the gas is introduced through the handle and burns at a number of fine holes on the upper surface of the blade (Fig. 56).

Fig. 55.

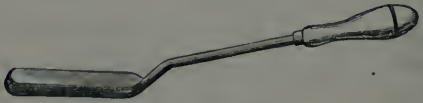
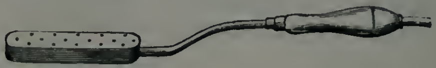


Fig. 56.



A piece of plaster is cut off the roll and melted, by means of the spatula, on a piece of brown paper, or in a small dish over Bunsen flame ; when fully melted it is transferred to the leather and spread evenly over the surface by rapid stokes of the spatula, taking especial care of the edges and corners. The spatula must not be heated too strongly, the least heat that will thoroughly melt the plaster being all that should be applied. Belladonna and menthol plasters especially should not be over-heated. If too hot it is also liable to penetrate and discolour the leather. Blisters are not heated at all, an ordinary steel spatula or the thumb being used to spread them. Now remove the shape, which should leave the leather quite clean, and trim the leather, leaving a margin proportionate to the size of the plaster, usually ¾ to 1¼ inch.

Plasters having an *adhesive* margin require two shapes, and soft soap must be used to prevent the second shape sticking to the plaster.

Ointments.—*Unguenta* (*ointments*), like the plasters, consist of an active medicine and a basis ; the latter is usually of a fatty or similar soft nature.

The chief object sought in the preparation of an ointment is perfect *smoothness*, then capability of keeping free from rancidity or other change.

Ointment Bases.—The bases most commonly employed are benzoated lard, prepared lard, soft paraffin, mixtures of soft and hard paraffin in varying proportions, wool fat (lanoline), or mixtures of these, with or without the addition of liquid oils and waxes.

Whenever the object desired is the absorption of the medicament, as in such ointments as belladonna and mercury, prepared and benzoated lard are to be prefered above all other bases, on account of their rapid absorption by the skin ; if, however, the object is rather to form a protective covering for a wound and to exert a purely local action, as in the case of boric acid ointment, a mixture of hard and soft paraffin is the most desirable basis ; when used in the proportion of 2 parts of soft to 1 part of hard paraffin the ointment is specially adapted for spreading upon lint, as it can be removed without leaving a quantity of ointment adhering to the wound. The soft paraffin used should melt between 35° and 40·5° C., and the hard paraffin at about

57·5° C.* In the Pharmacopœia several of the ointments are prepared with paraffins in different proportions; *e.g.* Ung. Eucalypti contains equal parts, on account of the solubility of the basis in oil of eucalyptus, which renders it much softer; Ung. Hydr. Oxid. Rub., Ung. Veratrinæ, and some others contain soft paraffin 3, hard paraffin 1. This is, no doubt, with the object of lowering the melting-point and promoting absorption, which is, however, *very imperfect* in all cases where the paraffins are used. One great advantage of the paraffin bases is their power of keeping almost indefinitely, but in the case of red oxide of mercury ointment this property is not completely fulfilled. Many ointments are made up with "simple ointment"—a mixture of benzoated lard 1½ parts, almond oil 1½ fl. parts, and white wax 1 part; this is fairly well absorbed, and forms a nice basis, which keeps well in some cases in which lard fails. Lanolin alone is not a good basis; its chief advantage is (when anhydrous) ready admixture with watery fluids, but the stickiness and imperfect absorption more than compensate for this; it is much improved by admixture with fatty bases or soft paraffin.

Methods of Preparation.—In the preparation of ointments three principal methods are usually followed:

1. *Incorporation by trituration.*—This is performed either by pestle and mortar, or upon a porcelain, earthenware, or slate slab by means of a pliable spatula, usually of vulcanite. The active drug is placed on the slab, with a small quantity of the basis, and the two thoroughly mixed by rubbing together; more of the basis is then added and incorporated, and finally the remainder, *e.g.* Ung. Gallæ and Ung. Hydrargyri. This simple treatment is sometimes insufficient; it is necessary to first dissolve the active principle in water, spirit, or other suitable solvent, and then proceed as above,

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* The melting-points of fats, waxes, and similar substances are best determined as follows:—Liquefy a few grains, and draw a little of the liquid into a capillary glass tube; allow it to cool, and, if possible, reserve it for some hours before proceeding farther. Now tie the capillary tube to the bulb of a thermometer, immerse the bulb and tube in a test-tube containing water, and itself immersed in a beaker of water; heat the latter gently; at the moment the opaque wax or fat becomes transparent, note the temperature. The solidifying point is commonly a few degrees below the melting-point.

e. g. Ung. Potassii Iodidi, Ung. Aconitinæ, or to soften such substances as extracts in a similar way, *e. g.* Ung. Belladonnæ (not so directed in the Pharmacopœia). In those cases in which a more or less gritty powder has to be incorporated, it is best to rub the powder *perfectly smooth* with a very little almond oil (or some of the melted basis in a hot mortar), and add the remainder of the basis gradually after softening it by a slight warmth, *e. g.* Ung. Hydrarg. Ammoniati, Ung. Antimonii Tartarati, Ung. Calaminæ.

2. *Fusion.*—By this method the basis is first brought to a uniformly fluid condition by the heat of a water-bath, and the medicament is either melted along with the basis (Ung. Elemi) or it is sprinkled or poured in (if liquid) as the mass cools (Ung. Eucalypti; Ung. Hydrarg. Oxidi Rubri). By either of these methods it is necessary with nearly all ointments to stir constantly whilst cooling until the ointment is quite thick, and cold, or nearly so, to prevent the settling of heavy powders, and especially to prevent the formation of little lumps of congealed basis; this is particularly the case with bases containing hard paraffin, wax, or spermaceti, these substances being liable to harden before the other constituents, on account of their higher solidifying point. It is best to stir the cooling ointment in a round-bottomed open dish (not a pot with straight sides), continually stirring down the ointment from the edges with the spatula.

A combination of the two methods of fusion and trituration is often desirable, as, for instance, in the case of Ung. Zinci. First melt the benzoated lard, rub the oxide of zinc quite smooth with a little of the melted fat in a hot mortar, and add this to the remainder of the fat, stirring constantly as it cools.

In some instances the drug is added to the melted basis and the heat continued until solution is effected, when the heat is withdrawn, and the mixture allowed to cool, stirring briskly to ensure the separation of the drug in the finest possible state of division, *e. g.* Ung. Chrysarobini; Ung. Iodoformi.

In all cases of fusion, the heat employed should not exceed that which is barely necessary to ensure liquefaction or solution; this is more especially necessary when volatile substances are present, *e.g.* Ung. Iodoformi; Ung. Hydragyri Compositum.

3. *Digestion and subsequent incorporation.*—In these cases the medicament is first digested (allowed to stand in contact) with one or all of the ingredients of the basis, either cold or hot, the liquid fat separated by straining and expression (if necessary), and then cooled as usual. The B. P. examples are Ung. Cantharidis, Sabinæ, Staphisagriæ.

All the ointments are included in this chapter for convenience.

The following is a list of the B. P. ointments arranged in order of strength; the student should prepare those marked *, and any others he may wish.

Name.	Basis.	Strength.	Method of preparation.	Note.
Unguentum Picis . .	Yellow wax	5 in 7	Melt wax, add tar, and stir constantly	
„ Conii . .	Lanolin	2 of succus in 1, with 10 grs. boric acid per oz.	Evaporation and trituration	1
„ Zinci Oleati .	Soft paraffin	1 in 2	Melt together, stir till cold	
„ Hydrargyri .	Lard 16, suet 1	1 in 2$\frac{1}{16}$	Trituration till free from visible globules	
„ Terebinthinæ	Lard 4, yellow wax 4, resin 1	1 in 2$\frac{1}{8}$	Fusion	2
* „ Hydrargyri Compositum	Olive oil 1, yellow wax 1	1 of Ung. Hydrarg. and $\frac{1}{4}$ of camph. in 2$\frac{1}{4}$	Do.	3
„ Staphisagriæ	Benzoated lard	1 in 2$\frac{1}{4}$ (about)	Digestion while hot and straining	4
* „ Sabinæ .	Benzoated lard 8, yellow wax 1$\frac{1}{2}$	1 in 2$\frac{1}{2}$	Do.	5
„ Hydrargyri Nitratis Dilutum	Soft paraffin	1 of Ung. Hydr. Nit. in 3	Trituration	6
„ Resinæ .	Simple ointment 8, yellow wax 2, almond oil 1	1 in 3$\frac{3}{4}$	Fusion and straining	
„ Antimonii Tartarati	Simple ointment	1 in 5	Trituration	
„ Elemi . .	Do.	1 in 5	Fusion and straining	
„ Eucalypti .	Soft paraffin 1, hard paraffin 1	1 in 5	Fusion and mixture	
„ Sulphuris .	Benzoated lard	1 in 5	Trituration	

Name.	Basis.	Strength.	Method of preparation.	Note.
*Unguentum Cetacei .	Almond oil 10, white wax 1, spermaceti 2½, benzoin ¼	1 in 5·4 (nearly)	Fusion and digestion	6
„ Calaminæ .	Benzoated lard	1 in 6	Trituration	
„ Glyc. Plumbi Subacetatis	Soft paraffin 3, hard paraffin 1	1 in 6⅓	Fusion and mixture	
„ Gallæ . .	Benzoated lard	1 in 6½	Trituration	
„ Hydrargyri Subchloridi	Do.	1 in 6½	Do.	
* „ Zinci . .	Do.	1 in 6½	Trituration and fusion	
„ Acidi Borici .	Soft paraffin 2, hard paraffin 1	1 in 7	Fusion and incorporation while hot	9
„ Cantharidis .	Olive oil 6, yellow wax 1	1 in 7 (nearly)	Digestion cold, straining, pressure, and fusion	7
* „ Hydrargyri Nitratis	Olive oil 2, lard 1	1 in 8 (nearly)	Fusion accompanied by chemical action	8
„ Hydrargyri Oxidi Rubri	Soft paraffin 3, hard paraffin 1	1 in 8 (nearly)	Fusion and trituration	9
„ Plumbi Carb.	Simple ointment	1 in 8	Trituration	
„ „ Iodidi	Do.	1 in 8	Do.	
* „ Potass. Iodidi	Benzoated lard	1 in 8¾ (nearly)	Solution and trituration	10
„ Creasoti .	Simple ointment	1 in 9 (nearly)	Trituration	
* „ Belladonnæ .	Benzoated lard	1 of alc. extract in 10	Trituration with little proof spirit	
„ Hamamelidis	Simple ointment	1 of fl. extract in 10	Trituration	
„ Hydrargyri Ammoniati	Do.	1 in 10	Do.	
* „ Iodoformi .	Benzoated lard	1 in 10	Solution by fusion, and assiduous stirring while cooling	
„ Gallæ c̄ Opio	Ointment of galls	1 in 14·6	Trituration	
„ Potassæ Sulphuratæ	Soft paraffin 3, hard paraffin 1	1 in 15½ (nearly)	Trituration and fusion	9
„ Sulphuris Iodidi	Do.	1 in 15½ (nearly)	Trituration	9
„ Acidi Carbolici	Soft paraffin 2, hard paraffin 1	1 in 19	Fusion	
„ Chrysarobini	Benzoated lard	1 in 25	Solution by fusion, and assidous stirring while cooling	
„ Acidi Salicylici	Soft paraffin 2, hard paraffin 1	1 in 28	Do.	
„ Hydrargyri Iodidi Rubri	Simple ointment	1 in 28·3	Trituration	

Name.	Basis.	Strength.	Method of preparation.	Note.
Unguentum Iodi .	Prepared lard and glycerine	1 in 31, with 1 of iodide of potass.	Solution and trituration	11
„ Plumbi Acetatis	Benzoated lard	1 in 37½	Trituration	
„ Aconitinæ .	Do.	1 in 59	Solution in S. V. R. 3½, and subsequent trituration	
„ Atropinæ .	Do.	1 in 59	Do.	
„ Veratrinæ .	Soft paraffin 3, hard paraffin 1	1 in 63	Fusion and trituration	9, 12

Note 1.—The hemlock juice is first evaporated to one eighth at a temperature not exceeding 60° C., the boric acid added, and finally the hydrous wool fat. (Anhydrous wool fat answers better, as some watery liquid separates on keeping, if made from the hydrous preparation.)

Note 2.—Dissolve the resin in the warm oil of turpentine, and add this to the wax and lard previously melted on a water-bath; stir till stiff.

Note 3.—Melt the wax with the oil, add the ointment of mercury, and, *when nearly cold*, the camphor in fine powder, stirring thoroughly.

Note 4.—Crush the seeds, add them to the lard previously melted over a water-bath, digest for two hours, then remove from the bath, strain through calico, and set aside to cool. The melted lard extracts oil and alkaloids from the seeds.

Note 5.—The savin tops (fresh) are digested for twenty minutes with the wax and lard previously melted, expressed through calico, and stirred while cooling.

Note 6.—This is made like benzoated lard (q. v.); it requires careful stirring to avoid lumps.

Note 7.—Digest the cantharides (slightly bruised, not in powder) in the oil for twelve hours, then heat on water-bath for fifteen minutes, strain through muslin and press, add the liquid to the wax, previously melted, and stir till cold.

Note 8.—This ointment is made by a special method, and its successful production depends upon the chemical action between the fats and the mercury solution being allowed to proceed to a certain degree, but not beyond it. The formula is as follows:—Mercury 1 part, nitric acid 3 fl. parts, dissolved by the aid of a little heat, then add it to lard 3¾ parts, and olive oil 8 fl. parts, melted together on a steam-bath, and both liquids at a temperature of nearly 93·4° C., mixing thoroughly; continue the heat until brisk action occurs, accompanied by much frothing and evolution of nitrous fumes; then stir till cold. If too much heat be applied, the ointment darkens in colour; if too little, the action continues after preparation, causing the ointment to "froth." See also P. J. (3), xiii, p. 364, November 4th, 1882.

Note 9.—In common with most of the ointments having soft and hard paraffins as a basis, it is preferable to first prepare the basis by melting the paraffins together, and allow to cool with constant stirring. This basis is kept as a stock, and the ointments prepared from it by simple trituration as described above, after slightly softening but not melting the basis by a gentle heat to facilitate admixture.

Note 10.—The iodide of potassium is dissolved together with one sixteenth its weight of carbonate of potassium in seven-eighths part of water, and the solution incorporated with the lard by trituration. The object of the alkali is to prevent liberation of iodine by the fatty acids, which object is but imperfectly attained.

Note 11.—The B. P. directs the iodine and iodide of potassium to be rubbed with $2\frac{1}{7}$ parts of glycerin and the prepared lard incorporated by trituration. The glycerin effects the solution of the iodine.

Note 12.—The veratrine is rubbed smooth with 7 parts of olive oil, added to the hard and soft paraffin previously melted and partially cooled, and incorporated by trituration.

Other Preparations by Fusion.—It now only remains to briefly direct attention to a few chemicals and pharmaceutical compounds in the preparation of which fusion is a most important operation.

The chemicals referred to are—

Antimonium Nigrum Purificatum.—Fuse the mineral in a Hessian crucible; siliceous matter separates, and may be removed by skimming.

Argenti Nitras (sticks). $3Ag_2 + 8HNO_3 = 6AgNO_3 + 4OH_2 + 2NO$.—Fuse in platinum or porcelain, and pour into cylindrical moulds.

Bismuthum Purificatum.—Crude bismuth frequently contains lead, copper, iron, silver, arsenic, antimony, tellurium, and selenium; these are removed by fusion at low redness with cyanide of potassium and sulphur, then with about 5 per cent. of a mixture of equal parts of dried carbonates of sodium and potassium.

Potassa Caustica and *Soda Caustica* are prepared by boiling down the respective solutions in a silver vessel until a drop solidifies on cooling, when it is run into cylindrical moulds. Silver is the only available metal which resists the action of fused caustic soda or potash.

Potassa Sulphurata.—Potassium carbonate, 2 parts, is dried, then mixed with 1 part of sublimed sulphur in a warm mortar, and *slowly* heated to dull redness in a Cornish or Hessian crucible. The fused mass is poured on to a cold flagstone, covered with a porcelain basin whilst cooling, and when cold broken up and put into well-stoppered bottles. The following equation expresses the main reaction: $3K_2CO_3 + 4S_2 = K_2S_2O_3 + 2K_2S_3 + 3CO_2$. When fresh it is liver-coloured, but by oxidation in the air it assumes a greenish and finally a pale greyish colour.

Potassii Acetas.—$K_2CO_3 + 2CH_3COOH = 2CH_3COOK + CO_2 + H_2O$. Prepared by solution, filtration, evaporation, and fusion in basin by gentle heat, about 250° C.

Potassii Bromidum and Sodii Bromidum.—Solution—$6KHO + 3Br_2 = KBrO_3 + 5KBr + 3OH_2$, evaporation, then by fusion, $2KBrO_3 + 3C_2 = 2KBr + 6CO$. Then dissolve in very small proportion of water (*lixiviation*) to separate salts from insoluble matter.

Potassii Cyanidum.—$K_4FeC_6N_6 = 4KCN + Fe_2C + N_2 + C$.

Potassii Ferrocyanidum.—$K_2CO_3 + 2C_2 + N_2$ by fusion $= 2KCN + 3CO$. The carbon and nitrogen are derived from organic matter, blood, hoofs, &c. Then by boiling $6KCN + Fe + OH_2 + O = K_4FeC_6N_6 + 2KHO$.

Potassii Iodidum and Sodii Iodidum.—Like bromides, but substituting iodine for bromine.

Potassii Permanganas.—$6KHO + KClO_3 + 3MnO_2 = 3K_2MnO_4 + KCl + 3OH_2$ by fusion, then by boiling $3K_2MnO_4 + 2OH_2 = K_2Mn_2O_8 + 4KHO + MnO_2$, or by chlorine $2K_2MnO_4 + Cl_2 = K_2Mn_2O_8 + 2KCl$.

Sodii Arsenias.—$As_2O_3 + 2NaNO_3 + Na_2CO_3 = Na_4As_2O_7 + N_2O_3 + CO_2$ by fusion, then by solution in water—$Na_4As_2O_7 + OH_2 = 2Na_2HAsO_4$.

Sodii Carbonas.—$2NaCl + H_2SO_4 = Na_2SO_4 + 2HCl$.
$Na_2SO_4 + C_2 = Na_2S + 2CO_2$.
$Na_2S + CaCO_3 = Na_2CO_3 + CaS$.

Sodii Valerianas.—$C_5H_{11}OH + O_2$ (from $K_2Cr_2O_7$ and H_2SO_4) $= C_4H_9COOH + OH_2$.

$2C_5H_{11}OH + O_2$ (from $K_2Cr_2O_7$ and H_2SO_4) $= C_4H_9COOC_5H_{11} + 2OH_2$.

$C_4H_9COOH + C_4H_9COOC_5H_{11} + 2NaHO = 2C_4H_9COONa + C_5H_{11}OH + OH_2$.

Evaporate and carefully fuse like potassium acetate.

Zinci Chloridum.—Prepared like Liquor Zinci Chloridi, the clear solution being evaporated until a little of the liquid solidifies on cooling; then poured into moulds, and when partially cooled transferred to well-stoppered bottles. It rapidly absorbs water from the air.

The following pharmaceutical preparations should be made by the student.

Argenti et Potassii Nitras (mitigated caustic).—Nitrate of silver 1 part, nitrate of potassium 2 parts. Powder the crystals, and fuse in a platinum or porcelain basin by a small Bunsen flame, and pour into suitable moulds. A small bougie mould (p. 144) answers well; it should be lubricated with soft paraffin. "Toughened caustic" is prepared similarly, but contains only 5 per cent. of nitrate of potassium.

Sulphuris Iodidum.—Iodine 4 parts, sublimed sulphur 1 part. Mix well, heat *gently* in a glass flask, the neck of which is closed by a plug of cotton wool, until uniform in colour, then melt, taking care to rinse down any sublimed iodine with the molten mass. Finally allow to cool, and remove by breaking the flask. It is not a definite chemical compound (see 'P. J.' [3], xvi, p. 583).

Questions on Chapter XIII.

1. What is meant by "fusion"? Describe the action of heat upon a fusible solid.
2. Describe the preparation of Suppositoria Plumbi Composita.
3. Describe the preparation of Adeps Præparatus and Adeps Benzoatus.
4. What is the strength of the official suppositories of morphine, iodoform, mercury, and carbolic acid?
5. Describe the preparation of Emplastrum Ammoniaci c̄ Hydrargyro.
6. What is the strength of the following plasters?—Opium, cantharides, belladonna, iodide of lead, menthol, and warm plaster.
7. How would you spread a plaster of belladonna for the breast, six inches in diameter?
8. Name the most important ointment bases, with notes as to their applications.
9. Describe the preparation of the ointments of oleate of zinc, stavesacre, boric acid, nitrate of mercury, red oxide of mercury, and chrysarobin.
10. For what objects are the following used?—Rectified spirit in aconitine and atropine ointments, water in iodide of potassium ointment, and glycerine in iodine ointment.
11. Give the strength of each of the following ointments:—Red iodide of mercury, aconitine, veratrine, galls and opium, conium, oxide of zinc, belladonna, salicylic acid, cantharides, mercury, iodine, and diluted nitrate of mercury.

12. Describe the preparation of mitigated caustic.

13. Write equations for the reactions occurring in the preparation of the following, separate equations if in distinct stages:—Potassii Iodidum, Argenti Nitras, Potassii Permanganas, Sodii Arsenias.

14. What quantity (theoretical) of carbonate of potassium and of acetic acid, B. P., will be required to produce 72 lbs. of acetate of potassium, and how much carbon dioxide will be evolved?

CHAPTER XIV

PRECIPITATION—SCALE PREPARATIONS—DIALYSIS

Precipitation.—It sometimes happens that when two clear solutions of different salts are mixed, the resulting mixture is no longer clear, a quantity of insoluble matter separates from the solution. We have already seen cases of this, *e. g.* the preparation of Liquor Sodæ Chlorinatæ. This insoluble matter is called a *precipitate,* and the process *precipitation ;* it is caused by the interchange of acidulous radicals amongst the bases present according to the law of Berthollet, which is as follows :

"When we cause two salts to react by means of a solvent, if in the course of double decomposition a new salt *can* be produced less soluble than those which we have mixed, this salt *will* be produced."

Coagulation.—Precipitation is also produced by other causes besides double decomposition of salts ; for instance, in order to remove albuminous matter from juices or watery extracts of dried plants boiling is frequently resorted to, whereby the albumen is precipitated. This is frequently termed *coagulation,* but is in reality only a form of precipitation.

Granulation.—Again, to remove mucilaginous substances from similar liquids spirit is added, whereby they are rendered insoluble ; the same process is sometimes applied to strong aqueous solutions of salts, as in the case of granulated sulphate of iron : the spirit causes the precipitation of the salt in a finely crystalline condition. This operation also has received the term *granulation,* which, however, is better reserved for the effervescent preparations alone.

Official Preparations.—The student should prepare the following :

Hydrargyri Iodidum Rubrum.

Take of—

Perchloride of mercury	4 parts.
Iodide of potassium	5 ,,
Boiling distilled water	80 ,,

Dissolve the perchloride of mercury in 60 parts, and the iodide of potassium in 20 parts of the water, and mix the solutions. When the liquid has cooled, decant from the precipitate, and having collected the latter on a calico strainer, wash it twice with cold distilled water, and finally dry it at a temperature not exceeding 100° C.

When first precipitated mercuric iodide is yellowish, but it rapidly changes to brilliant scarlet; the proportions ordered must be rigidly adhered to, as the precipitate is soluble in excess of either salt. The drying may be carried out on the small scale in a soup plate over a water-bath, or on trays in the drying room. $HgCl_2 + 2KI = HgI_2 + 2KCl$.

Ferri Phosphas.

Take of—

Sulphate of iron	3 parts.
Phosphate of sodium	$2\frac{3}{4}$,,
Bicarbonate of sodium	$\frac{3}{4}$ part.
Boiling distilled water	q. s.

Dissolve the sulphate of iron in 30 parts of the water, and the phosphate of sodium in a similar quantity of water. When each solution has cooled to between 37·8° and 54·4° C. add the latter to the former, pouring in also a solution of the bicarbonate of sodium in a little distilled water. Mix thoroughly. Transfer the precipitate to a calico filter, and wash it with hot distilled water till the filtrate ceases to give a precipitate with chloride of barium. Finally dry the precipitate at a temperature not exceeding 120° F. (48·9° C.).

The great object sought is to prevent oxidation of the ferrous phosphate, to attain which all the water used should be boiled (to expel dissolved oxygen) and then partly cooled for use, and the whole operation should be carried out quickly. It is best to dissolve the sulphate of iron in *hot*, not *boiling* water. The bicarbonate of sodium is added to neutralise phosphoric acid, which is set free as shown in the following equations:

$$6Na_2HPO_4 + 6FeSO_4 = 2Fe_32PO_4 + 6Na_2SO_4 + 2H_3PO_4.$$
$$2H_3PO_4 + 6NaHCO_3 = 2Na_3PO_4 + 6OH_2 + 6CO_2.$$
$$2Na_3PO_4 + 3FeSO_4 = Fe_32PO_4 + 3Na_2SO_4.$$

Chloride of barium is used to indicate whether the washings are free from sulphate of sodium; if not a white precipitate occurs.

Liquor Ferri Acetatis Fortior.

Take of—

Solution of persulphate of iron	5 fl. parts.
Solution of ammonia	a sufficiency.
Glacial acetic acid	3 fl. parts.
Distilled water	a sufficiency.

Mix the ammonia (about 8 fl. parts) with 20 fl. parts of distilled water; to this add the solution of persulphate of iron, previously diluted with about 20 fl. parts of distilled water; stir the whole thoroughly, taking care that the ammonia is finally in slight excess, as indicated by the odour. Let stand two hours, put it on a calico filter, drain off liquor, and wash with distilled water till the filtrate is free from sulphate of ammonium; let it drain, tie the precipitate up in the cloth, then remove superfluous moisture by pressure; add the precipitate to the glacial acetic acid, and stir occasionally till dissolved; make up to 10 fl. parts with distilled water; finally allow any insoluble matter to subside, and decant the clear solution. Sp. gr., when finished, 1·127.

The following reactions occur:

$$Fe_2 3SO_4 + 6NH_4HO = Fe_2 6HO + 3\,(NH_4)_2SO_4.$$
$$Fe_2 6HO + 6CH_3COOH = 6(CH_3COO)Fe_2 + 6OH_2.$$

Ferri Sulphas Granulata.

Take of—

Iron wire	4 parts.
Sulphuric acid	4 fl. parts.
Distilled water	30 ,,
Rectified spirit	8 ,,

Pour the water on the iron placed in a porcelain capsule, add the sulphuric acid, and when the disengagement of gas has nearly ceased boil for ten minutes, and then filter the solution into a jar containing the spirit, stirring the mixture so that the salt shall separate in minute granular crystals. Let these, deprived by decantation of adhering liquid, be transferred on filtering paper to porous tiles, and dried by exposure to the atmosphere. The following reaction occurs: $Fe + H_2SO_4 = FeSO_4 + H_2$.

The presence of the hydrogen prevents oxidation to ferric salt; if the boiling were left till all the iron had dissolved, much ferric salt would be produced. The stirring is necessary, or the heavy solution would sink to the bottom of the spirit without mixing to any appreciable extent, and deposit the sulphate of iron in *large* crystals.

Official Salts.—Precipitates are preferably prepared in hot solutions, as they are then more easily washed, pressed, and dried, being denser; but in some cases it is not advisable to use hot water, *e. g.* iodide of lead, which is dissolved, and to a less extent red iodide of mercury. Sometimes the precipitate is formed in cold solutions, the whole being afterwards boiled to cause aggregation of the particles of precipitate; in others the whole is carried out in the cold, *e. g.* oxide of silver. They are usually dried at or slightly below 100° C. on a water-bath or in hot air chamber; but in some cases a lower temperature is employed, *e. g.* carbonate and subnitrate of bismuth (65·5° C.), arseniate and phosphate of iron (48·9° C.),—in the former cases to prevent loss of one molecule of water, in the latter to avoid undue oxidation to ferric salts.

The following are the official compounds:

Precipitated cold and washed cold:

Argenti Oxidum, $2AgNO_3 + Ca2HO = Ag_2O + Ca2NO_3 + OH_2$. Dry at 100° C.

Plumbi Iodidum, $Pb2NO_3 + 2KI = PbI_2 + 2KNO_3$. Dry at gentle heat.

Ferri Peroxidum Hydratum, $Fe_23SO_4 + 6NaHO = Fe_26HO + 3Na_2SO_4$. Dry at 100° C.

By drying, $Fe_26HO = Fe_2O_22HO + 2OH_2$.

Hydrargyrum Ammoniatum, $HgCl_2 + 2NH_4HO = NH_2HgCl + NH_4Cl + 2OH_2$. Dry at 100° C.

Bismuthi Subnitras, $Bi3NO_3 + 2OH_2 = BiONO_3,OH_2 + 2HNO_3$. Dry at or below 65·5° C. (Some Bi remains in solution.)

Bismuthi Carbonas, $2Bi3NO_3 + 2N_3H_{11}C_2O_5 + 2OH_2 = Bi_2O_2CO_3,OH_2 + 6NH_4NO_3 + 3CO_2$. Dry at or below 65·5° C.

Sodii Bicarbonas, $Na_2CO_3 + CO_2 + OH_2 = 2NaHCO_3$.

Sulphur Præcipitatum, $3Ca2HO + 6S_2 = 2CaS_5 + CaS_2O_3 + 3OH_2$. By boiling with water.
$2CaS_5 + CaS_2O_3 + 6HCl = 6S_2 + 3CaCl_2 + 3OH_2$. Dry at 49° C.

Precipitated hot or warm and washed cold:

Hydrargyri Iodidum Rubrum, see above.

Hydrargyri Oxidum Flavum, $HgCl_2 + 2NaHO = HgO + 2NaCl + OH_2$. Dry near 100° C.

Antimonium Sulphuratum,

$$\left.\begin{array}{r}2Sb_2S_3 + S_2 + 6NaHO = 2Na_3SbS_4 + Sb_2O_3 + 3OH_2 \\ Sb_2O_3 + 6NaHO = 2Na_3SbO_3 + 3OH_2\end{array}\right\}\text{; then}$$

$2Na_3SbS_4 + 2Na_3SbO_3 + 6H_2SO_4 = Sb_2S_5 + Sb_2O_3 + 6Na_2SO_4 + 3SH_2 + 3OH_2$. Dry at 100° C.

Precipitated hot or boiling and washed hot:

Ferri Arsenias, $3FeSO_4 + 2Na_2HAsO_4 + 2NaHCO_3 = Fe_32AsO_4 + 3Na_2SO_4 + 2CO_2 + 2OH_2$. Dry at 49° C.

Ferri Phosphas, see above.

Calcii Carbonas Præcipitata, $CaCl_2 + Na_2CO_3 = CaCO_3 + + 2NaCl$. Dry at 100° C.

Calcii Phosphas, $Ca_32PO_4 + 4HCl = CaH_42PO_4 + 2CaCl_2 + 2OH_2$; then $CaH_42PO_4 + 2CaCl_2 + 4NH_4HO = Ca_32PO_4 + 4NH_4Cl + 4OH_2$. Dry at 100° C.

Solution mixed, then boiled and washed:

Bismuthi Citras, $Bi3NO_3 + Na_3C_6H_5O_7 = BiC_6H_5O_7 + 3NaNO_3$. Dry below 100° C.

Magnesii Carbonas levis, $4MgSO_4 + 4Na_2CO_3 + 5OH_2 = (MgCO_3)_3Mg(HO)_2,4OH_2 + 4Na_2SO_4 + CO_2$. Boiled 15 minutes, washed and dried below 100° C.

Magnesii Carbonas ponderosa, ditto, ditto. Stronger solutions are used, the whole *evaporated* to dryness and heated on a sand-bath, then washed and dried as for the light variety.

Zinci Carbonas, $3ZnSO_4 + 3Na_2CO_3 + 3OH_2 = 3Na_2SO_4 + ZnCO_3(Zn2HO)_2,OH_2 + 2CO_2$. Boiled 15 minutes, dried below 100° C.

Also—

Antimonii Oxidum, $12SbCl_3 + 15OH_2 = 2SbCl_3,5Sb_2O_3 + 30HCl$. Precipitate; then $2SbCl_3,5Sb_2O_3 + 3Na_2CO_3 = 6Sb_2O_3 + 6NaCl + 3CO_2$. Dried below 100° C.

Bismuthi Oxidum, $2(BiONO_3,OH_2) + 2NaHO = Bi_2O_3 + 2NaNO_3 + 3OH_2$. Boiled 5 minutes, washed, dried below 100° C.

Lotions and other Pharmaceutical Preparations.—There are also several distinctly pharmaceutical preparations dependent upon precipitation. Two of these have already been noticed; the others are—

Lotio Hydrargyri Flava.—Perchloride of mercury, 1 part; lime water, 243 fl. parts. $HgCl_2 + Ca2HO = HgO + CaCl_2 + OH_2$.

Lotio Hydrargyri Nigra.—Subchloride of mercury, 1 part; lime water, 146 fl. parts. $2HgCl + Ca2HO = Hg_2O + CaCl_2 + OH_2$. The salts are simply agitated with the solution of lime.

Liquor Magnesii Carbonatis (fluid magnesia; see also p. 101).—Mix freshly precipitated carbonate of magnesium with water, and force into the mixture pure washed CO_2 under pressure of three atmospheres; after twenty-four hours filter, and pass in more CO_2; finally, securely close the bottle containing the solution.

**Liquor Morphinæ Bimeconatis.*—Hydrochlorate of morphine, 1 part; solution of ammonia, q. s.; meconic acid, $\frac{2}{3}$ part; rectified spirit, 25 fl. parts; distilled water, q. s. Dissolve the morphine salt in about 15 parts of the water, then add solution of ammonia in very slight excess; cool, filter, wash the precipitate with cold water till free from chloride, drain, mix the precipitate with enough distilled water to produce 75 parts, then add the meconic acid dissolved in the spirit, filter through white paper *free from iron*, and make up volume with distilled water to 100 fl. parts. Iron gives a red coloration with meconic acid. = about $1\frac{1}{4}$ parts in 100 fl. parts, or $\frac{3}{4}$ part of morphine.

Injectio Morphinæ Hypodermica.—Hydrochlorate of morphine, 9·2 parts; solution of ammonia, acetic acid, distilled water, of each q. s. The precipitation is carried out as above, the morphine mixed with about 40 parts of the water, kept warm, and the acetic acid added drop by drop until the morphine is dissolved and the solution is very faintly acid to litmus paper; finally make up the volume to 87·5 fl. parts, and filter. = 1 grain of acetate of morphine in 10 minims. This preparation is liable to deposit a basic acetate; a small proportion of glycerine and S. V. R. prevents this ('P. J.' [3], xxii, p. 848).

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* To be prepared by the student.

Syrupus Ferri Phosphatis.—Granulated sulphate of iron, 224 grains; phosphate of sodium, 200 grains; bicarbonate of sodium, 56 grains; concentrated phosphoric acid, 1¼ fl. ounces; refined sugar, 8 ounces; distilled water, 8 fl. ounces. Precipitate and wash phosphate of iron (see p. 160); transfer the precipitate to a mortar and triturate it with the acid; when dissolved, filter; add the sugar, and dissolve without heat; finally add water, if necessary, to produce 12 fl. ounces. It contains very nearly one grain of anhydrous phosphate of iron in each fl. drachm. Sp. gr. about 1·305.

The author prefers to make this syrup by dissolving iron wire, 46 grains, in phosphoric acid, 1 oz. 3 fl. drs., mixed with an equal volume of water; boiling to expel an unpleasant odour, and adding the filtered solution to 9 fl. ounces of simple syrup, finally adjusting to 12 fl. ounces with distilled water. Less acid (1 fl. oz.) makes a more pleasant syrup.

**Ferri Carbonas Saccharata.*—Sulphate of iron, 2 parts; carbonate of ammonium, 1¼ parts; boiling distilled water, 320 parts; refined sugar, 1 part. Dissolve the salts each in 80 parts of the water, and mix the solutions. Allow the precipitate to subside in a covered jar, and siphon off the supernatant liquid. Wash with the remainder of the water by decantation. Collect on calico, press, and triturate with the sugar. Finally, dry the mixture at or below 100° C.

$$3FeSO_4 + 2N_3H_{11}C_2O_5 + OH_2 = 3FeCO_3 + 3(NH_4)_2SO_4 + CO_2.$$

When freshly prepared it contains about 50 per cent. of ferrous carbonate (reckoned as anhydrous), but this amount is gradually lessened by oxidation on keeping, ferric oxide being produced. On this account the preparation should not be finely powdered, but left in small lumps about as large as white mustard seeds. The B. P. requires at least 33·3 per cent. real $FeCO_3$.

Precipitation forms an important step in the production of most of the so-called "scale" preparations and of dialysed iron, which we will now proceed to describe.

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* To be prepared by the student.

Scale Preparations.

Scaling.—Scale preparations owe their peculiar "flaky" character to the fact that they are dried in very thin layers on glass plates. Their production depends upon the fact that citric, tartaric, and some other organic acids form soluble compounds with iron and some other metals, which are not precipitated by ammonia, or by soda or potash unless boiled; consequently the metallic salt is in a basic condition. This essential operation of *scaling* is carried out as follows:—Having reduced the solution by evaporation to a syrup, it is thinly painted on glass plates by means of a brush; the plates are then arranged in suitable horizontal frames in the drying room or closet, which should be provided with a good draught of dry air, and maintained at about 27° C., certainly not above 38° C., as directed in B. P. The "paint" will soon be seen to crack in all directions, after which it begins to rise off the plates in places. When thoroughly dry, evidenced by general looseness of the "scales," the plates are removed, the scales detached while still warm by tapping, and if necessary gently scraping with a spatula; the small scales and dust sifted out by means of a coarse sieve, and the larger ones quickly transferred to well-corked bottles. The dust, &c., may be re-dissolved and re-scaled. Although this is the actual operation of scaling, great care must be exercised throughout the whole process of preparation of the liquid for scaling if good results are to be obtained. For instance, in the preparation of scale compounds of iron, which are very numerous, the following points must be attended to:

1. The production of insoluble oxysulphate or oxyhydrate of iron must be avoided by adding *the iron solution to the ammonia in excess,* and washing *thoroughly* and *rapidly* by decantation.

2. The temperature of the liquid during evaporation should not exceed 82° C., but the vessels in which it is carried on should be very shallow, so as to effect the evaporation as rapidly as possible. These precautions are necessary to prevent reduction to ferrous salt, which is especially liable to occur with tartrates.

The following are the scale preparations of the B. P.:

Beberinæ Sulphas, *vide* Chap. XXI.

Bismuthi et Ammonii Citras.—Evaporate Liq. Bismuthi et Ammon. Citratis over a water-bath to the consistence of a thin syrup, and plate as usual. *It does not form good scales;* if containing full B. P. quantity of bismuth they are very small and dull-looking.

Ferri et Ammonii Citras.—

"Take of—

Solution of persulphate of iron	10 fl. parts or a sufficiency.
Solution of ammonia	23 ,, ,,
Citric acid	4 parts.
Distilled water	a sufficiency.

"Mix 16 fl. parts of the solution of ammonia with 40 parts of distilled water, and to this add gradually the solution of persulphate of iron, previously diluted with 40 parts of distilled water, stirring them constantly and briskly, and taking care that ammonia is, even finally, in slight excess, as indicated by the odour. Let the mixture stand for two hours, stirring it occasionally; then put it on a calico filter, and when the liquid has drained away wash the precipitated ferric hydrate with distilled water until that which passes through the filter ceases to give a precipitate with chloride of barium. Dissolve the citric acid in 4 parts of distilled water, and having applied the heat of a water-bath, add the ferric hydrate, previously well drained, and stir them together until nearly the whole of the hydrate has dissolved, or until the citric acid is saturated with ferric hydrate (prepared, if necessary, from more of the solution of persulphate of iron). Let the solution cool, then add 5½ parts of solution of ammonia. Filter through flannel, adding some distilled water if necessary; evaporate to the consistence of syrup, the presence of a very slight excess of ammonia being maintained, and dry in thin layers on flat porcelain or glass plates at a temperature not exceeding 100° F. (37·8° C.). Remove the dry salt in flakes, and keep it in a stoppered bottle."—B. P.

Note.—The direction to *saturate* the citric acid with ferric hydrate is inconsistent with the B. P. test of about 30 per cent. oxide of iron; if saturated, the percentage would be nearer 36 per cent.

Ferri et Quininæ Citras (to be made by the student).—

“Take of—

Solution of persulphate of iron	4½ fl. ounces.
Sulphate of quinine	1 ounce.
Diluted sulphuric acid	1½ fl. ounces.
Citric acid	3 ounces 30 grains.
Solution of ammonia Distilled water } of each	a sufficiency.

“Precipitate the iron solution as usual, using 8 fl. ounces of the solution of ammonia; while washing this precipitate mix the sulphate of quinine with 8 ounces of distilled water, add the sulphuric acid, and when the salt is dissolved precipitate the quinine with a slight excess of solution of ammonia. Collect the precipitate on a filter, and wash it with a pint and a half of distilled water. Dissolve the citric acid in 5 ounces of distilled water, and, having applied the heat of a water-bath, add the ferric hydrate, previously well drained, stir them together, and when the hydrate has dissolved add the precipitated quinine, continuing the agitation until this also has dissolved. Let the solution cool, then add in small quantities at a time 12 fl. drachms of solution of ammonia diluted with 2 fl. ounces of distilled water, stirring the solution briskly, and allowing the quinine which separates with each addition of ammonia to dissolve before the next addition is made. Filter the solution, evaporate it to the consistence of a thin syrup, then dry it in thin layers on flat porcelain or glass plates at a temperature of 100° F. (37·8° C.).”—B. P. = 15 per cent. quinine.

Ferrum Tartaratum.—

Take of—

Solution of persulphate of iron	6 fl. parts.
Solution of ammonia	11 „
Acid tartrate of potassium	2 parts.
Distilled water	a sufficiency.

Precipitate and wash ferric hydrate as usual, and when drained mix it intimately with the acid tartrate of potassium; let stand twenty-four hours; then, having applied heat not exceeding 60° C., add gradually a pint of distilled water, and stir constantly until nothing more will dissolve. Filter, and evaporate at a temperature not exceeding 60° C. to the consistence of syrup, and scale as usual.

Unofficial Scale Compounds.—There are numerous non-official scale preparations, the chief of which are—

Ferri Citras (U. S. Pharm.)	26 % ferric oxide.
„ Pyrophosphas	Ferri Cit. 9 parts, Sodii Pyrophos. 10 parts.
„ et Ammonii Tartras (U. S. Pharm.)	25 % Fe_2O_3.
„ et Strychninæ Citras (U. S. Pharm.)	1 % strychnine.
„ et Quininæ et Strychninæ Citras	1 % strychnine, 15 % quinine.
„ et Cinchonidinæ Citras	15 % cinchonidine.
„ et Cinchoninæ Citras	15 % cinchonine.
Potassii Boro-tartras	20 % boric acid, 80 % cream of tartar.

Dialysis.

Liquid Diffusion.—When a solution having a sp. gr. greater than that of water is introduced into a cylindrical vessel, and water carefully poured over it, so as not to mix the liquids, the substance dissolved in the lower liquid will slowly rise into the supernatant water, although the liquids may remain undisturbed and the temperature unchanged. This phenomenon is known as *liquid diffusion.* Different substances diffuse with unequal velocities; thus crystalline substances, such as sodium chloride and potassium iodide, diffuse much more rapidly than amorphous bodies, such as gum or gelatine. On this account bodies of great diffusibility have been called *crystalloids,* while substances of low diffusive power are termed *colloids* (*i. e.* gelatine-like).

This property of the unequal diffusion of substances has been turned to account in the separation of crystalloids from colloids when existing together in the same solution, both in pharmacy and in analysis. With the latter we have nothing to do here, but an illustration of the former occurs in the B. P., Liq. Ferri Dialysatus.

It has been found that the diffusion of liquids takes place with greater rapidity if a porous diaphragm be interposed between the two liquids; this is no doubt due to the influence of capillary attraction.

Osmose.—This important property of diffusion through a porous diaphragm has been called *osmose.* Osmose usually takes place more rapidly in one direction than the other; that is, the more crystalloid body passes through the diaphragm in one direction in greater quantity than the less crystalloid passes in the opposite direction. In time a state of equilibrium will be reached, consequently it is necessary to frequently change the liquid on one side of the diaphragm. This process of osmose is the cause of the absorption of water through the extremities of rootlets; it also assists the rise of sap throughout the cellular tissue of the plant, and plays an important part in the assimilation of nutriment in the digestive organs of animals.

Dialysis.—In the operation of *dialysis* the mixed solution is placed in a shallow vessel (Fig. 57), open at the upper end, but having a sheet of parchment paper securely tied over the lower end; this is supported on a stand (Fig. 58) in a larger vessel containing water (Fig. 59), or suspended as in Fig. 60; the crystalloid passes outwards into the water, leaving the colloid within the *dialyser.* The water in the outer vessel must be frequently changed, or an automatic supply arranged to keep the level uniform if rapid dialysis is required.

FIG. 57.

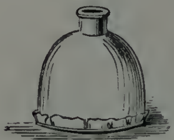


FIG. 58.

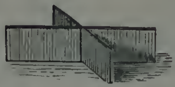


FIG. 59.

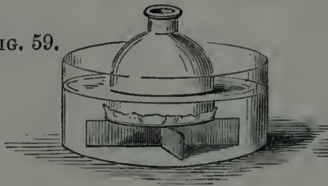
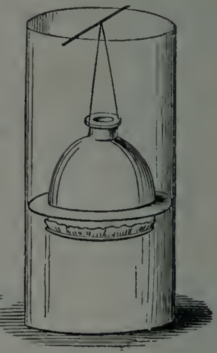


FIG. 60.



Liq. Ferri Dialysatus.—The B. P. Liq. Ferri Dialysatus is a solution of highly basic *ferric oxychloride,* or *chloroxide of iron,* and is prepared as follows:

Take of—

Strong solution of perchloride of iron . .	7 fl. parts.
Solution of ammonia Distilled water } of each . . .	a sufficiency.

Having precipitated the ferric hydrate from 6 fl. parts of the solution of perchloride of iron as usual, wash and press; add the precipitate to the remainder of the solution of perchloride of iron, stir thoroughly, and allow to stand in a warm place until the liquid is clear or nearly so; filter if necessary, and introduce into the dialyser; then dialyse as usual until the solution on the dialyser is almost tasteless. The resulting fluid should measure 28 fl. parts, and have a sp. gr. 1·047.

By this means almost the whole of the ferric chloride is removed, the liquid remaining on the dialyser consisting practically of a solution of ferric hydrate with a trace of chloride.

Precipitation is often resorted to as a means of purifying solutions of chemicals, as in the official Liq. Zinci Chloridi, from which lead and iron are removed by means of chlorine and carbonate of zinc. Thus—

$$2FeCl_2+Cl_2=Fe_2Cl_6.$$

$$Fe_2Cl_6+ZnCO_3(Zn2HO)_2OH_2=Fe_26HO+3ZnCl_2+CO_2.$$

Similarly calcium chloride, which is finally dried at 204° C.

Questions on Chapter XIV.

1. What is Berthollet's law of precipitation?

2. Distinguish between precipitation and coagulation.

3. Describe the preparation of granulated sulphate of iron, yellow oxide of mercury, heavy carbonate of magnesium, syrup of phosphate of iron, and injection of morphine.

4. Give equations of the reactions occurring in the preparation of saccharated carbonate of iron, ammoniated mercury, precipitated sulphur, sulphurated antimony, arseniate of iron, phosphate of calcium, and carbonate of zinc.

5. Give the formulæ for black and yellow mercurial lotions; what compounds are present in the final preparations?

6. What is the strength of each of the following?—Syrup of phosphate of iron, solution of bimeconate of morphine, and fluid magnesia.

7. Describe the preparation of citrate of iron and quinine, giving reasons for each stage of the process.

8. Define "osmose;" describe its practical application in pharmacy.

9. How much nitrate of lead will be required for the preparation of one third of a kilogramme of iodide of lead?

CHAPTER XV

CRYSTALLISATION—GRANULATION.

Crystalline and Amorphous.—When subjected to the proper conditions the great majority of substances are capable of assuming regular geometrical forms, *e. g.* alum in octahedra, sodium chloride in cubes.

This act is called *crystallisation;* the geometrical solids are called *crystals,* and are said to be *crystalline.* Those substances which are incapable of crystallisation are said to be *amorphous.* Many substances assume two different crystalline forms when subjected to different conditions; these are said to be *dimorphous.* An example presents itself in sulphur, which may occur in octahedra or in prisms. Substances possessing the same crystalline forms are said to be *isomorphous.*

Crystals frequently cleave in certain directions; the flat surfaces thus produced are the *planes* or *faces,* the lines where two planes meet are the *edges;* the inclination of two edges is a *plane angle,* the point where three or more edges meet is a *solid angle.*

The various crystalline forms have been classified into six classes or systems, according to the number, relative length, and direction of the *axes,** which are imaginary lines passing through the geometrical centre and joining the angles or the centres of the planes.

Methods of Crystallisation.—There are numerous methods by which crystals may be obtained; the most important are the following.

1. By solution in water, alcohol, or some other solvent; if the compound be more soluble in the hot than the cold liquid a hot saturated solution is prepared, when the crystals separate out on cooling; or a weak solution is evaporated until a film or pellicle forms on the surface,

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* For a general description of this part of the subject the student should consult a work on crystallography, or Fownes' "Inorganic Chemistry."

and then left to cool as before. The liquor from which crystals have separated is known as the *mother-liquor*. If, however, there be little difference between the solubility of the compound in the hot or cold solvent, a solution is left to evaporate spontaneously, either in the air or in a partial vacuum; or the substance may be obtained in very small and irregular crystals by simply evaporating the solution to dryness during continuous stirring. This last is the official method of obtaining Potass. Carbonas, Potass. Citras, and some others; it is also known as *granulation*.

2. By fusion and slow cooling. This is particularly useful in the case of bismuth and some other metals.

3. By sublimation, a good instance of which occurs in iodine.

Whichever method of preparation be adopted, it may be taken as a general rule that the *more slowly* the operation is carried out, the *more perfect* will be the resulting crystals.

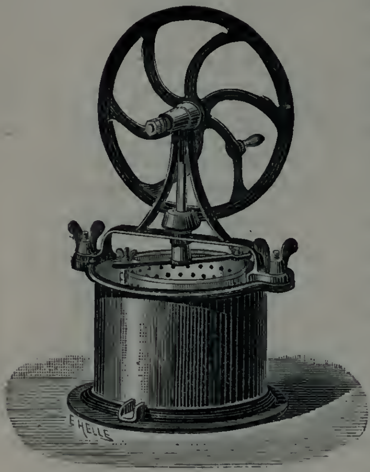
Water of Crystallisation.—When crystallised from water, it frequently happens that the crystals retain a certain proportion of the solvent in a state of combination; the combination is, however, not nearly so intimate as that which obtains in the case of the union of an acid and a base, for example; for the water can in almost every case be readily removed by such simple means as the application of a gentle heat, the essential properties of the chemical remaining intact, although the crystalline character is either changed or entirely destroyed. Such a loose combination is termed *molecular*, as distinct from *atomic* combination.

The water so retained is called *water of crystallisation*, and the crystals are said to be *hydrous*. Crystals which contain no water of crystallisation are said to be *anhydrous*. The same salt may crystallise from water with varying proportions of water of crystallisation, *e.g.* sodium bromide crystallised from cold water has the formula $NaBr,2H_2O$; when obtained from water at or above 60° C. it is, however, anhydrous. Sodium carbonate from cold water contains ten molecules, from boiling water one molecule, and at intermediate temperatures eight or five molecules of water. Some crystals rapidly absorb water from the atmosphere at ordinary temperatures, becoming damp; these are said to be *deliquescent.* Others, again, lose moisture, becoming dry and powdery on the surface; these are called *efflorescent.*

Alcohol and other solvents are also occasionally retained in a similar manner by crystals deposited from those liquids.

Washing and Drying of Crystals.—Crystals obtained from solutions require to be dried, and frequently need purification from impurities present in the mother-liquor, either by simple washing with cold water (or alcohol, &c.), or by re-solution and recrystallisation from a fresh quantity of solvent. After the final crystallisation the crystals may be taken out of the mother-liquor, or the mother-liquor siphoned off, the crystals transferred to a funnel or other suitable vessel to drain, washed by pouring cold water (or alcohol, &c.) in small quantities at a time over the contents of the funnel, and dried. The method of drying varies with the nature of the crystals: if anhydrous, they may usually be introduced at once into the drying cupboard or room on trays; but if hydrous they must be dried without heat, or at such a temperature as not to drive off any of the water of crystallisation. This is frequently done by simply exposing the crystals to a good draught in layers upon bibulous paper or on porous tiles; but by far the best plan is to remove all superfluous water by centrifugal force,—that is, the force by virtue of which loose particles are driven from the centre to the circumference of a revolving body. To accomplish this the crystals are introduced into a porous cylinder which is enclosed in an earthenware, metal, or wooden box; the cylinder is provided with suitable gearing, whereby it can be rapidly revolved. By the rapid revolution any moisture is quickly driven to the outside, and passing through the small holes of the cylinder is collected in the outer box. By this means

Fig. 61.



crystals can be rapidly and almost perfectly dried in a short time, and with very little exposure to the air. Fig. 61 represents a small centrifugal machine, worked by hand.

Official Compounds.—Crystallisation forms a very important part of the process of manufacture of a great number of Pharmacopœial preparations—mostly, however, chemicals and definite principles obtained from plants. These latter will be considered in a later article, whilst the former are treated in works on chemistry; those which have not previously been mentioned in other sections, such as "fusion" (KI, NaBr, &c.) and sublimation ($HgCl_2$, &c.), will now be described by equations.

Name of Compound.	*Equation.*
Acidum Boricum	$Na_2B_4O_7,10OH_2+H_2SO_4=4H_3BO_3+Na_2SO_4+5OH_2$.
„ Chromicum	$K_2CrO_4CrO_3+H_2SO_4=2CrO_3+2KHSO_4+OH_2$.
„ Citricum	$2H_3C_6H_5O_7+3CaCO_3=Ca_32C_6H_5O_7+3CO_2+3OH_2$. $Ca_32C_6H_5O_7+3H_2SO_4=2H_3C_6H_5O_7+3CaSO_4$.
„ Gallicum	See Chap. XXI.
„ Meconicum	See „
„ Salicylicum	$C_6H_4(OH)COOCH_3+KHO=CH_3OH+C_6H_4(OH)COOK$ $C_6H_4(OH)COOK+HCl=C_6H_4(OH)COOH+KCl$ * $C_6H_5OH+NaHO=C_6H_5ONa+OH_2$ $C_6H_5ONa+CO_2=C_6H_4(OH)COONa$ $C_6H_4(OH)COONa+HCl=C_6H_4(OH)COOH+NaCl$ †
„ Tartaricum	$2KHC_4H_4O_6+CaCO_3=CaC_4H_4O_6+K_2C_4H_4O_6+CO_2+OH_2$. $K_2C_4H_4O_6+CaCl_2=CaC_4H_4O_6+2KCl$. $2CaC_4H_4O_6+2H_2SO_4=$ ‡$2H_2C_4H_4O_6+2CaSO_4$.
Acetanilidum	$C_6H_5NH_2+CH_3COOH=C_6H_5,NH,COCH_3+OH_2$.
Alumen	$Al_23SO_4+(NH_4)_2SO_4+24OH_2=Al_23SO_4(NH_4)_2SO_4,24OH_2$.

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* Natural from oil of winter-green and birch.

† Artificial from carbolic acid.

‡ Constitutional formula $\left.\begin{matrix}CH(OH)COOH\\CH(OH)COOH\end{matrix}\right\}$

Name of Compound.	*Equation.*
Ammonii Benzoas	$C_6H_5COOH + NH_4HO^* = C_6H_5COONH_4, OH_2$.
,, Phosphas	$2NH_4HO+H_3PO_4{}^*=(NH_4)_2HPO_4+2OH_2$.
Antimonium Tartaratum	$Sb_2O_3 + 2KHC_4H_4O_6 = 2(KSbOC_4H_4O_6),OH_2$.
Apomorphinæ Hydrochloras	See Chap. XXI.
Argenti Nitras	$3Ag_2+8HNO_3=6AgNO_3+2NO+4OH_2$.
Atropina	See Chap. XXI.
Borax	$4H_3BO_3+Na_2CO_3+4OH_2=Na_2B_4O_7,10H_2O+CO_2$; *also native.*
Caffeina	See Chap. XXI.
Cinchonidinæ Sulphas	See ,,
Cinchoninæ Sulphas	See ,,
Cocainæ Hydrochloras	See ,,
Codeina	See ,,
Cupri Nitras	$3Cu_2+16HNO_3+10OH_2=6(Cu2NO_3,3OH_2)+4NO$.
,, Sulphas	$CuO + H_2SO_4 + 4OH_2 = CuSO_4,5OH_2$.
Elaterinum	See Chap. XXI.
Ferri Sulphas	$Fe + H_2SO_4 + 7OH_2 = FeSO_4,7OH_2 + H_2$.
,, ,, Granulata	See page 161.
Homatropinæ Hydrobromas	See Chap. XXI.
Iodoform	$C_2H_5HO+3K_2CO_3+4I_2= CHI_3+HCOOK+5KI+2OH_2+3CO_2$.
Lithii Citras	$3L_2CO_3+2H_3C_6H_5O_7+5OH_2=3CO_2+2(L_3C_6H_5O_7,4OH_2)$.
Magnesii Sulphas	$MgCO_3+H_2SO_4 + 6OH_2 = MgSO_4,7OH_2+CO_2$.
Morphinæ Acetas, &c.	See Chap. XXI.
Picrotoxinum	See ,,
Pilocarpinæ Nitras	See ,,
Plumbi Acetas	$PbO+2CH_3COOH + 2OH_2{}^\dagger=(CH_3COO)_2Pb,3OH_2$.
,, Nitras	$PbO + 2HNO_3 = Pb(NO_3)_2 + OH_2$.
Potassii Bicarbonas	$K_2CO_3 + CO_2 + OH_2 = 2KHCO_3$.
,, Bromidum, KI, &c.	See page 156.
,, Chloras	$\left\{\begin{array}{l} 6Ca(HO)_2+6Cl_2=3(CaCl_2,CaCl_2O_2)+6OH_2. \\ 3(CaCl_2,CaCl_2O_2) = Ca2ClO_3 + 5CaCl_2 \text{ (by boiling).} \\ Ca2ClO_3 + 2KCl = CaCl_2 + 2KClO_3. \end{array}\right.$
,, Nitras	Recrystallised from native salt.

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* The water must contain a slight excess of free ammonia, or the product will be *acid.*

† The water must contain a slight excess of free acetic acid, or the product will be slightly basic.

Name of Compound.	*Equation.*
Potassii Sulphas	$2KNO_3+H_2SO_4=K_2SO_4+2HNO_3$.
„ Tartras	$2KHC_4H_4O_6+K_2CO_3+OH_2=2(K_2C_4H_4O_6,OH_2)+CO_2$.
„ „ Acida	Recrystallised from argol.
Quininæ Hydrochloras	See Chap. XXI.
„ Sulphas	
Saccharum Lactis	From whey of milk.
„ Purificatum	From juice of sugar-cane, &c.
Salicinum	See Chap. XXI.
Santoninum	See „
Soda Tartarata	$2KHC_4H_4O_6+Na_2CO_3+7OH_2=CO_2+2(KNaC_4H_4O_6,4OH_2)$.
Sodii Arsenias (NaBr, &c.)	See page 156.
„ Nitras	By recrystallisation from native salt.
„ Nitris	$2NaNO_3=2NaNO_2+O_2$.
„ Phosphas	$2Na_2CO_3+CaH_4(PO_4)_2+23OH_2=2(Na_2HPO_4 12OH_2)+CaCO_3+CO_2$.
„ Sulphas	$2NaHSO_4+Na_2CO_3+19OH_2=2(Na_2SO_4,10OH_2)+CO_2$.
„ Sulphis	$Na_2CO_3+SO_2+7OH_2=Na_2SO_3,7OH_2+CO_2$.
„ Sulphocarbolas	$C_6H_5OH+H_2SO_4=C_6H_4(OH)SO_3H+OH_2$. $2C_6H_4(OH)SO_3H+Na_2CO_3+3OH_2=2(C_6H_4ONaSO_3H,2OH_2)+CO_2$.
Strychnina	See Chap. XXI.
Zinci Acetas	$ZnCO_3(Zn2HO)_2,OH_2+6CH_3COOH=3\left\{\begin{matrix}CH_3,COO\\CH_3,COO\end{matrix}\right\}Zn2OH_2+CO_2$.
„ Sulphas	$Zn+H_2SO_4+7OH_2=ZnSO_4,7OH_2+H_2$.
„ Sulphocarbolas	$2C_6H_4(OH)SO_3H+ZnO=Zn(C_6H_4OSO_3H)_2,OH_2$.
„ Valerianas	$ZnSO_4+2C_4H_9COONa=\left\{\begin{matrix}C_4H_9COO\\C_4H_9COO\end{matrix}\right\}Zn+Na_2SO_4$.

Granulation.

Granulation is the process whereby solids are obtained in the state of a nearly uniform crystalline or semi-crystalline, coarse powder. It is generally applied to three distinct classes of preparations, two of which have already been mentioned.

1. Finely crystalline salts obtained by precipitation from solution by means of another liquid in which the salts are insoluble, *e. g.* Ferri Sulphas Granulata (p. 161).

2. Finely crystalline or semi-crystalline salts obtained by evaporation of solutions during constant stirring, *e. g.* Potassii Carbonas.

3. Semi-crystalline compounds possessing the property of effervescing (*i. e.* bubbling rapidly, due to evolution of gas) when added to water, and prepared by a process to be presently described.

Official Compounds.—*Class* 1.—The only B. P. example is Ferri Sulphas Granulata.

Class 2.—The following are the official examples:

Name.	*Equation.*
Ammonii Bromidum	$NH_4HO + HBr = NH_4Br + OH_2$.
,, Chloridum	$NH_4HO + HCl = NH_4Cl + OH_2$.
Arsenii Iodidum	$As_2O_3 + 6HI = 2AsI_3 + 3OH_2$.
Atropinæ Sulphas	$2C_{17}H_{23}NO_3 + H_2SO_4 = (C_{17}H_{23}NO_3)_2H_2SO_4$. Note 1.
Calcii Hypophosphis	$3Ca2HO + 2P_4 + 6OH_2 = 3Ca2PH_2O_2 + 2PH_3$.
Potassii Carbonas	Recrystallised from pearlashes.
,, Citras	$3K_2CO_3 + 2H_3C_6H_5O_7 = 2K_3C_6H_5O_7 + 3CO_2 + 3OH_2$.
Sodii Benzoas	$Na_2CO_3 + 2C_6H_5COOH = 2C_6H_5COONa + CO_2 + OH_2$.
,, Bromidum	See page 156.
,, Chloridum	Recrystallised from native salt.
,, Hypophosphis	$3NaHO + P_4 + 3OH_2 = 3NaPH_2O_2 + PH_3$.
,, Iodidum	See page 156.
,, Salicylas	$2C_6H_4(OH)COOH + Na_2CO_3 = CO_2 + 2C_6H_4(OH)COONa + OH$.

Note 1.—Exact neutrality must be attained, and the solution evaporated at or below 38° C., to avoid injury to the product.

Class 3.—The following are official:

Magnesii Sulphas effervescens = 50 per cent. $MgSO_4, 7H_2O$.

Sodii Citro-tartras effervescens.

Sodii Phosphas effervescens = 50 per cent. $Na_2HPO_4, 12H_2O$.

Sodii Sulphas effervescens = 50 per cent. $Na_2SO_4, 10H_2O$.

The student should prepare the following according to the official directions:

“ *Sodii Citro-tartras Effervescens.*

“ Take of—

Bicarbonate of sodium, in powder	17 parts.
Tartaric acid, in powder	9 „
Citric acid, in powder	6 „
Refined sugar, in powder	5 „

“ Mix the powders thoroughly, place them in a dish or pan of suitable form heated to between 200° and 220° F. (93·3° and 104·4° C.), and when the particles of powder begin to aggregate, stir them assiduously until they assume a granular form, then by means of suitable sieves separate the granules of uniform and most convenient size, and preserve the preparation in well-closed bottles.”

All effervescent granular preparations contain these ingredients as a basis. When salts containing much water of crystallisation have to be so prepared, they must first be dried to such an extent that they do not liquefy at the temperature employed, otherwise the whole will become a pasty mass. The other three official preparations are cases in point, and require the following proportions of water to be removed before granulation: Mag. Sulph. 23 per cent., Sodii Phosphas 60 per cent., Sodii Sulphas 56 per cent. of the weight of the crystalline salt.

For small quantities a shallow evaporating basin of thin porcelain, placed over a steam-bath, answers admirably; but on the large scale, large flat-bottomed pans heated by steam are employed, and the powders stirred about by means of rakes. In either case care must be taken to break down the larger masses as they form, the heat being turned on rapidly *at first*, whereby the salts aggregate into small pasty lumps; the heat is then lessened until the granules are dried, or if large pasty lumps be formed they may be divided by rubbing through sieves whilst soft. The whole must be kept stirred till the granules are quite white and *hard*, when they are obtained of uniform size by sifting. The sieves used may be No. 6 and No. 20; all which passes through the latter being rejected, and that which is retained by the former broken down. When made strictly according to B. P. directions a considerable proportion is thus rejected.

Sodii Citro-tartras is similar to the popular preparation known as citrate of magnesia, but is less sweet.

There is a vast number of these effervescent preparations in the market, among the most important being, in addition to the above—

Citrate of lithium	2 or 5	grs. per drm.
Citrate of caffeine	2 or 5	,,
Antipyrin	4 or 10	,,
Phenacetin	5 or 10	,,
Ammonio-citrate of bismuth	5	,,
Citrate of potassium	10	,,
Bromide of potassium	5	,,
Citrate of iron and quinine	5	,,
Citrate of iron and ammonium	5	,,
Hydrobromate of caffeine	2	,,

Granulated zinc and tin are prepared by pouring a thin stream of the molten metal into cold water.

Questions on Chapter XV.

1. Define crystalline, amorphous, dimorphous, isomorphous, mother-liquor, water of crystallisation.
2. What qualities of crystals are expressed by the following terms?—Anhydrous, efflorescent, deliquescent.
3. Describe three methods of obtaining crystals, and give two examples of each.
4. Give equations for the production of ammonium benzoate, copper nitrate, lithium citrate, sodium phosphate, and sodium sulphocarbolate.
5. Note any peculiarities attached to the crystallisation of ammonium benzoate, copper nitrate, lead acetate, sodium bromide, atropine sulphate.
6. Describe three methods of granulation, and give examples of each.
7. Describe the preparation of sodium benzoate.
8. Write equations for the production of sodium hypophosphite, arsenium iodide, and sodium salicylate.
9. Give names and strength of the B. P. effervescent granular preparations.

CHAPTER XVI

EXTRACTION OF DRUGS

This is the most important operation in the whole range of pharmacy. It is obvious that, no matter how carefully the other operations of concentration, filtration, &c., be conducted, the preparation will be unsatisfactory unless the active constituents be extracted by suitable methods, employing suitable solvents. The object sought is to remove from a crude drug the whole of the active constituent or constituents, whilst rejecting those substances which are inert, or which would render the resulting preparation liable to decomposition.

Solvents for Extraction.—The first thing to be considered is naturally, what is the most suitable solvent? The following rules will serve as a guide to the student, although special drugs may require special solvents.

1. Drugs whose activity depends upon the presence of a volatile oil or resin should be extracted by means of a strong spirituous solvent, *e. g.* asafœtida, myrrh, sumbul.

2. Drugs whose activity depends upon the presence of an alkaloid or glucoside are *usually* most satisfactorily exhausted by a weaker spirit* (proof spirit; rectified spirit 3 parts, water 1 part; or rectified spirit and water, equal parts), *e. g.* belladonna, aconite.

3. Drugs whose activity depends upon the presence of mucilaginous substances can only be represented by aqueous solvents, *e. g.* cetraria, linseed.

4. Drugs whose activity depends upon astringent principles, such as tannin, may be commonly represented by either water or proof spirit, *e. g.* catechu, matico.

Methods of Extraction.—Numerous methods are employed to secure extraction, but all of these may be included under two general heads, viz. maceration and percolation.

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* A great many of these drugs can also be fully extracted by aqueous solvents, but mixed with a larger proportion of inert matter, *e.g.* gentian, calumba.

Maceration.—The process of *maceration* consists in allowing the solvent (called the *menstruum*) to stand in contact with the drug, previously reduced to a suitable degree of comminution for a certain period of time, the whole being agitated or stirred occasionally. At the expiration of the specified period the liquid is strained off, the residual drug (called the *marc*) subjected to pressure, or in some cases washed by affusion with more of the menstruum to make up to a specified volume. The following special terms have been applied to various modifications of the method of maceration.

1. *Simple maceration.*—This method is officially employed for the preparation of most of the wines, many of the tinctures and extracts, besides some other preparations. It is carried out as described above, the time and menstruum varying with the nature of the drug and class of preparation required.

Wines—Vina.—The wines form a class of preparations made by extracting the desired constituents of the drugs by means of wine. Sherry is employed except in the case of wine of citrate of iron and quinine wine, for which orange wine is used: these two are not prepared by maceration.

The general directions for the preparation of the official wines are represented by the following, which should be made by the student.

" *Vinum Colchici.*—Colchicum corm sliced, dried, and reduced to No. 20 powder, 1 part; sherry, 5 fl. parts. Macerate the colchicum in the wine for seven days in a closed vessel with occasional agitation, press and strain through calico; then add sufficient sherry to make one pint."

The points requiring comment are—

(1) " No. 20 powder." Directions for the comminution of drugs have already been given (Chap. III).

(2) " Seven days." The specified period should be rigidly adhered to, as carelessness in this respect tends to produce want of uniformity in preparations. Whenever a wine or other preparation is set on to macerate it should be clearly labelled, somewhat as follows, to avoid error in this particular:

VINUM COLCHICI.
Set on . . . 2/II/93.
To come off . . 9/II/93.
To make 2 pints.

(3) "Occasional agitation." The common mode of following this is to briskly agitate night and morning, which is sufficient; or the same object can be attained by enclosing the drug loosely in a muslin bag suspended just below the surface of the menstruum, contained in a very wide-mouthed vessel. The menstruum near the top thus becomes loaded with the soluble matter of the drug; its sp. gr. is thereby increased, and the heavier liquid falls to the bottom, to be replaced by the lighter. This constitutes an automatic "agitation." At the end of the seven days the bag with its contents is removed from the liquid and pressed.

(4) "Press the marc." The methods of expression have been previously described (Chap IX).

(5) "Strain and filter." The filtration is usually easily accomplished by a simple paper filter in the case of tinctures and wines; some of the latter, however, may be *brightened* by the addition of a little pumice or silica previous to filtration, *e. g.* Vin. Colchici, Vin. Rhei.

The following are the wines of the Pharmacopœia:

Name.	Formula.	Method of preparation.
Vinum Aloës . .	Soc. aloes 1½ oz.; cardam. seeds and ginger, of each 80 grs.; sherry 2 pints	Maceration.
,, Antimoniale .	Tartarated antimony 2 grs. in 1 fl. oz.	Solution (see page 69).
,, Colchici. .	1 of root in 5	Maceration. Note 1.
,, Ferri . .	1 of iron wire in 20	Maceration for 30 days. Note 2.
,, ,, Citratis .	8 grs. citrate of iron in the oz.	Solution (see page 69).
,, Ipecacuanhæ	1 of root in 20	Maceration, &c. (see p. 228).
,, Opii . .	1 of extract, with cinnamon and cloves of each $\frac{1}{6}$, in 20	Maceration.
,, Quininæ. .	1 gr. sulphate quinine and 1½ grs. citric acid in 1 oz.	Solution (see page 69).
,, Rhei . .	1½ oz. rhubarb and 60 grs. canella in 1 pint	Maceration.

Note 1.—If finely powdered, filtration is almost impossible owing to the abundance of starch in the corm.

Note 2.—This preparation contains about ¼ to ⅓ grain *iron* in 100 fl. grains when finished; it should not be allowed to stand beyond the specified time, as it begins to deposit. The iron is only partially immersed in order to promote oxidation; the acidity of the wine causes solution of a small quantity of iron as tartrate and acetate.

Tinctures (see p. 201).—The tinctures are a very numerous class of preparations, obtained by treating the drugs with spirit, so as to extract the constituents which are soluble in that liquid. The spirit employed may be of any desired strength, but the British Pharmacopœia prescribes only proof spirit, which contains nearly 50 per cent. by weight of absolute alcohol, and rectified spirit 56 o. p., containing 84 per cent. by weight of absolute alcohol.

Ammoniated tinctures are sometimes prepared with aromatic spirit of ammonia, or with mixtures of proof or rectified spirit with solution of ammonia (sp. gr. 959 or 891).

Ethereal tinctures.—The ethereal tincture of lobelia of the B. P. is prepared with spirit of ether, which is usually to be understood to be the menstruum required for ethereal tinctures, although a class of preparations intended for outward application exists which are made with ether.

It would be far better if the term "linimentum" or "pigmentum" were employed to designate such spirituous preparations as are primarily intended for outward application, reserving the name "tinctura" for liquids administered chiefly internally: *Tinctura Arnicæ* would then become *Linimentum Arnicæ; Tinctura Iodi* would be *Pigmentum* (or *Linimentum*) *Iodi dilutum.*

Many of the tinctures of the Pharmacopœia are prepared by maceration; but their consideration is reserved until the whole of the class is described together, when the subject of *percolation* has been reviewed. The remarks given under "wines" respecting the *time of maceration,* &c., apply equally to tinctures; one point, however, it is necessary to mention, viz. the fineness of the powders should be greater than for wines, as the spirit strength of the menstrua employed is greater, and the drugs will not absorb strong alcoholic liquors so readily as more aqueous ones.

The subjects of extracts and syrups are also reserved for the same reason.

2. *Digestion.*—This is nothing more than maceration at a temperature somewhat higher than the common temperature of the atmosphere. It is frequently employed. We have already had occasion to note several instances of its use among the ointments, viz. Ung. Cantharidis, in which digestion for fifteen minutes in oil placed in boiling water is preceded by cold maceration for twelve hours; Ung. Cetacei, and Adeps Benzoatus, in which the benzoin is digested in the liquefied fat for two hours; Ung. Staphisagriæ, the seeds digested two hours in lard melted over a water-bath; and Ung. Sabinæ, in which the tops are digested for twenty minutes in a molten mixture of benzoated lard and yellow wax. In addition to these we have Acetum Cantharidis, Charta Epispastica, Syrupus Sennæ, and several extracts, for which last see Chap. XIX.

The Chartæ (papers) of the Pharmacopœia are two in number, viz. Charta Epispastica and Charta Sinapis; only the former of these is prepared by digestion.

Charta Epispastica.

Take of—

White wax	16 parts.
Spermaceti	6 ,,
Olive oil	8 fl. parts.
Resin	3 parts.
Canada balsam	1 part.
Cantharides, in powder	4 parts.
Distilled water	24 fl. parts.

Digest all the ingredients except the Canada balsam in a water-bath for two hours, stirring them constantly; then strain and separate the plaster from the watery liquid. Mix the Canada balsam with the plaster melted in a shallow vessel, and pass strips of paper over the surface of the hot liquid, so that one side of the paper shall receive a thin coating of plaster.

It may be convenient to employ paper ruled, so as to indicate divisions, each of which is one square inch.

Note.—The Canada balsam is not heated with the other ingredients because its volatile oil would be dissipated to a great extent.

Water is employed in order to aid the separation of the residual cantharides from the oily mass.

For Charta Sinapis 1 part of mustard is mixed with 2 fl. parts of gutta-percha solution, and cartridge-paper passed over the surface so as to become thinly coated with the mixture; it is subsequently dried by exposure to the air.

3. *Infusion.*—The operation of *infusion* differs from maceration and digestion in this particular, that whereas in the two latter operations the temperature of the mixture is kept as regularly uniform as possible during the whole period, subject only to the variations due to changes in the temperature of the atmosphere or surrounding medium; in the operation of *infusion* water or other solvent at a certain temperature is added to the drug, and the mixture allowed to cool during a specified period, after which the marc is separated by straining.

In the preparation of infusions five points demand our attention: (1) the menstruum; (2) temperature; (3) condition of the drug; (4) the vessel in which the operation is conducted; (5) the time of infusing.

(1) *The menstruum.*—This is in all cases distilled water; *distilled* water presents advantages over spring water on account of the salts, especially calcium and magnesium salts, which are commonly present in the latter, which often, especially in the case of drugs containing tannin, modify the resulting preparation.

(2) *Temperature.*—In most cases boiling water is used, because the extraction of the drug is more rapid and more perfect; there are, however, *four* official exceptions, viz. infusions of calumba and quassia, for which cold water, and chiretta and cusparia, for which water at 120° F. (49° C.), is employed. In the case of calumba cold water is used because the drug contains much starch, which would give a mucilaginous and inelegant preparation; whilst in the other three cases infusions prepared with boiling water possess a more nauseous bitterness than the official ones: the active principles are also somewhat sensitive to heat.

(3) *Condition of the drug.*—Water being the solvent, and usually hot, it is unnecessary to reduce the drugs to a fine

powder, and in some cases positively injurious, *e. g.* buchu and calumba, on account of the presence of much mucilage or starch, the former of which would swell and impede straining; the latter would either almost entirely prevent it, or would pass through the strainer and spoil the appearance of resulting infusion. Some are directed to be used whole, viz. chamomiles, hop, digitalis, senna, and linseed: in the first two because no advantage is gained by comminution; in digitalis and senna on account of the mucilage, which in these cases is valueless; and in linseed because the mucilage (for which it is required) resides in the testa, and is readily extracted without crushing. For similar reasons others are to be "cut small," viz. orange, calumba, chiretta, jaborandi, and matico, "sliced" gentian and rhubarb; "bruised," buchu, cloves, bearberry, and valerian. Quassia is in "chips;" rose petals "broken up," that is the conglomerated petals separated from one another; and ergot "crushed:" this is to avoid undue removal of oil, which is present in large quantity. Catechu and cusso are in coarse powder, the former being readily exhausted in this condition, the latter softened. The remaining drugs require more careful comminution, as they are more resistant to the action of water; cascarilla, serpentary, and senega being in No. 20, and cinchona, cusparia, and rhatany in No. 40 powder.

Fig. 62.

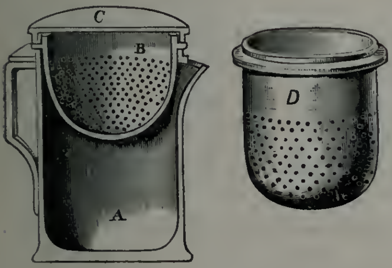
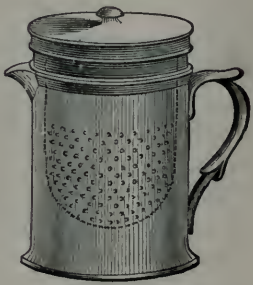


Fig. 62 a.



(4) *The vessel.*—The B. P. simply directs "a covered vessel." This is all that is necessary provided the contents are stirred occasionally during infusion, but a better form is Squire's infusion pot. The accompanying illustrations

(Figs. 62 and 62 A) explain its construction. The drug is placed in the colander B, and most of the water which is poured over it runs through into the outer vessel A. The colander keeps the drug in the upper part of the liquid, consequently in the hottest and least saturated portion. No stirring is needed, as there will be automatic circulation caused by the greater density acquired as the liquid in the colander becomes charged with soluble matter. This apparatus is, however, scarcely suitable for those drugs which are used in No. 40 powder. In such cases either the colander should be lined with fine muslin or calico, or an ordinary covered vessel used with frequent stirring.

(5) *Time.*—The time is of great importance, and should be rigidly adhered to, as in some cases the result is much modified by neglect; *e. g.* if infused much beyond fifteen minutes infusion of chamomile becomes a nauseous emetic instead of an aromatic bitter, whilst simple and compound infusions of orange become much impaired in flavour. In the B. P. of 1867 in many cases the time ordered was much longer, but the shorter time now adopted scarcely affects the strength of the resulting preparations, and is a great convenience for dispensers. See a paper by the author, 'Pharm. Journ.' (iii), xvii, p. 388.

To prepare an infusion with boiling water proceed as follows:—Boil more than the required quantity of water in a kettle, and meanwhile weigh out the drugs on paper. Pour some of the boiling water into the infusing vessel to thoroughly warm it, and throw this out; then introduce the drugs and quickly weigh. Put the required weights into the scale-pan, and then add the boiling water till counterpoised; cover closely and set aside, stirring occasionally if a colander be not used. At the expiration of the prescribed time pour the contents of the pot on a muslin strainer and allow to drain; do *not* add water to restore original volume.

The following is a tabular arrangement of the official infusions in order of strength; two at least to be made by the student.

Name.	Strength.	Time.	Remarks.	Note.
Infusum Digitalis	1 in 156	15 minutes	...	1
" Gentianæ Comp.	1 in 40	Half an hour	Also contains orange peel 1, fresh lemon peel 2	
" Quassiæ	"	"	Cold water	
" Aurantii Comp.	"	15 minutes	Also contains fresh lemon peel ½, cloves ¼	
" Caryophylli	"	Half an hour		
" Chiratæ	"	"	Water at 120° F. (48·9° C.)	
" Ergotæ	"	"		
" Rhei	"	"		
" Rosæ Acidum	"	"	Also contains diluted sulphuric acid ½ fl. part	2
" Serpentariæ	"	"		
" Valerianæ	"	1 hour		
" Lini	1 in 30 (nearly)	2 hours	Also contains liquorice root ⅓	3
" Catechu	1 in 27½ (nearly)	Half an hour	Also contains cinnamon about 1 in 150	4
" Anthemidis	1 in 20	15 minutes		
" Aurantii	"	"		
" Buchu	"	Half an hour		
" Calumbæ	"	"	Cold water	
" Jaborandi	"	"		
" Krameriæ	"	"		
" Maticæ	"	"		
" Senegæ	"	"		5
" Cinchonæ Acidum	"	1 hour	Also contains aromatic sulphuric acid ¼ fl. part	6
" Cuspariæ	"	"	Water at 120° F. (48·9° C.)	
" Lupuli	"	"		
" Uvæ Ursi	"	"		
" Cusso	1 in 16	15 minutes	Not strained	7
" Cascarillæ	1 in 10	Half an hour		
" Sennæ	"	"	Also contains ginger $\frac{1}{16}$	8

Note 1.—J. W. England has shown that cold water is preferable, as heat is injurious to the active principles of digitalis (see 'Pharm. Journ.' [iii], xx, p. 186).

Note 2.—Sulphuric acid is added to develop the colour and give a pleasant acidity.

Note 3.—Liquorice added to flavour, two hours allowed to thoroughly soak the testa.

Note 4.—Cinnamon added as an aromatic stomachic.

Note 5.—J. R. Hill has shown that this preparation deposits sapogenin, due to decomposition of saponin. Cold or warm water would be preferable (see 'Pharm. Journ.' [iii], xix, p. 811).

Note 6.—Sulphuric acid aids solution of the quinine and other active principles, the bulk of which would otherwise remain in the marc.

Note 7.—Not strained because active principle is insoluble in water.

Note 8.—Ginger added as aromatic to prevent griping.

The operation of infusion is officially applied to the exhaustion of several drugs for the production of galenical preparations, *e.g.* extract of aloes, warming plaster, &c.; also of syrups of hemidesmus, lemon, poppy, red poppy, and rose; these are treated under their respective series.

4. *Decoction.*—This operation consists of boiling water in contact with the drug, either for a specified time or until the volume of the liquor is reduced to a certain degree. The former is obviously the more rational method, as the length of time required to boil off a given quantity of water must vary according to the intensity of heat employed, as well as other factors.

The operation of decoction is employed in the preparation of the decoctions and some of the extracts, viz. Ext. Anthem., Ext. Cascar. Sagrad. Liq., Ext. Rhamni Frang. Liq., and combined with infusion in Ext. Gentianæ and Hæmatoxyli; also of Syrupus Tolutanus. The process of decoction is not applicable to the extraction of aromatic drugs, as the boiling would remove the volatile oils upon which their aroma depends; in fact, it should only be used in those cases in which the drugs are close and difficult of exhaustion by other means.

The following are examples of B. P. decoctions, and should be prepared by the student.

Decoctum Cinchonæ.

Take of—

Red cinchona bark in No. 20 powder .	1¼ parts.
Distilled water	20 parts or q. s.

Boil for *ten* minutes in a covered vessel. Strain the decoction when cold,* and pour as much distilled water over the contents of the strainer as will make the strained product measure one pint.

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* All decoctions are strained whilst *hot* unless otherwise stated.

Decoctum Granati Radicis.

Take of—

Pomegranate root bark, sliced . . .	2 parts.
Distilled water	40 „

Boil down to 20 fl. parts and strain, making the strained product up to 20 fl. parts if necessary by pouring distilled water over the contents of the strainer.

In order to determine the volume of the boiling liquid it is not convenient to pour the hot decoction into a measure, but the following plan may be adopted:—Before introducing the drug into the saucepan, which should be enamelled, introduce 20 parts of water and measure the depth of this by means of any convenient piece of wood or enamelled iron; if wood, it should have been thoroughly boiled with water previously. The wood is marked at the surface of the water, and this measure may be used to gauge the depth and consequently the volume of the boiling decoction.

The following are the B. P. decoctions:

Name.	Time of boiling.	Strength.	Division.	Note.
Decoctum Aloës Compositum	5 minutes	Ext. Socot. aloes 1; myrrh, saffron, and K_2CO_3, of each $\frac{1}{2}$; ext. liquorice 4; comp. tinct. cardam. 30; water to 100	Coarse powder	1
„ Cetrariæ .	10 minutes	1 in 20	Whole	2
„ Cinchonæ .	„	1 in 16	No. 20 powder	3
„ Hæmatoxyli	„	1 in 20, with $\frac{1}{8}$ cinnamon	In chips	4
„ Papaveris .	„	About 1 in 10 when completed (15 used)	Bruised	5
„ Quercûs .	„	1 in 16	Do.	
„ Sarsæ . .	„	About 1 in 8 when finished (12 used)	Cut transversely	6
„ „ Compositum	„	About 1 in 8, with $\frac{1}{10}$ of sassafras, guaiacum chips, and dried liquorice, and $\frac{1}{20}$ of mezereon bark (12 used)	Do.	6
„ Scoparii .	„	1 of dried tops in 20	Whole	
„ Taraxaci .	„	1 in 20	Sliced and bruised	
„ Pareiræ .	15 minutes	1 in 16	No. 20 powder	
„ Hordei .	20 minutes	About 1 in 10 (15 used)	Whole	2
„ Granati Rad.	To one half	1 in 10	Sliced	

Note 1.—Boil the extract of aloes, myrrh, Pot. Carb., and extract of liquorice with 40 parts of water for five minutes, then add the saffron, and allow the whole to cool; add the tincture of cardamoms, and, covering the vessel closely, allow the ingredients to macerate for two hours; finally, strain through flannel, pouring as much distilled water over the contents of the strainer as will make the strained product measure 100 fl. parts. The carbonate of potassium assists the solution of the myrrh; the saffron is not subjected to the boiling, as its aroma would thereby become impaired; the tincture is added when the aqueous liquid is *cold* to avoid loss of spirit by evaporation; the final maceration for two hours allows of the deposition of a part of the mucilaginous matter thrown out by the spirit.

Note 2.—Before preparing the decoction the drug is first washed with *cold* water to remove impurities.

Note 3.—The reason for allowing this decoction to cool before straining is to permit the deposition of a compound of the alkaloids with cincho-tannic acid, which is soluble in hot but not in cold water. It is removed to avoid unsightliness, as the strength is not materially reduced thereby, the quantity being but small. Dr B. H. Paul has shown that the drug is by no means fully exhausted, from 60 to 84 per cent. of the alkaloids being left in the marc.

Note 4.—To avoid loss of aroma the cinnamon is added at the end of the boiling.

Note 5.—For this preparation (which is almost exclusively employed for external use) the whole of the poppy capsules, including seeds, are bruised On the contrary, the seeds are removed before preparing the extract and syrup.

Note 6.—Previous to ebullition the solid ingredients of these two decoctions are digested in boiling water for an hour; this is to thoroughly swell and soak the hard woody parts.

Questions on Chapter XVI.

1. What is meant by the "extraction" or exhaustion of a drug?
2. Distinguish infusion, decoction, maceration, digestion.
3. Describe generally the principles which would guide you in the selection of solvents for the extraction of drugs.
4. Define menstruum, marc, decoction.
5. Describe minutely the preparation of wine of rhubarb, giving reasons for the various steps of the process.
6. Which of the "vina" are prepared by simple maceration?
7. Give (*a*) the strength, and (*b*) any practical notes on the following:—Vinum Antimoniale, Vinum Opii, Vinum Quininæ, and Vinum Ferri.
8. Describe accurately the preparation of any official infusion.
9. Give reasons for the following:—(*a*) Use of cold water for infusions of calumba and quassia; (*b*) water at 49° C. for infusions of chiretta and cusparia; (*c*) use of buchu and bearberry simply "bruised;" and (*d*) use of sulphuric acid in infusions of cinchona and rose.

10. Which of the infusions are allowed to stand fifteen minutes, and why? Which for one hour?

11. Describe the preparation of decoction of sarsaparilla and compound decoction of aloes, with notes on the various points.

12. Give the strength of the following:—Infusions of cinchona, digitalis, quassia, senna, jaborandi, and valerian, and decoctions of broom, logwood, and pareira.

CHAPTER XVII

EXTRACTION OF DRUGS (*continued*)

Percolation or Displacement.

Percolation or Displacement.—The object of *percolation* or *displacement* is to subject the drug to the action of a continually renewed solvent by making provision for the removal of the menstruum as it becomes charged with the soluble constituents of the drug, to be followed by a fresh supply of the unused solvent.

The term *percolation* (from *per*, through; and *colo*, I strain through) refers to the menstruum *flowing through* the powdered drug; *displacement* expresses the following up of the charged menstruum by fresh solvent.

The Percolator.—The vessel in which percolation is carried out is called the *percolator;* it is usually constructed of glass, earthenware, copper, tinned copper, tinned or galvanised iron, or enamelled iron. Generally speaking, glass or earthenware is to be preferred: the former has the great advantage of transparency, thus allowing the operator to observe the progress of the operation. Metal percolators, especially galvanised iron, must be used with caution, as many drugs will cause solution or corrosion of the surface.

Form of Percolator.—The form of the percolator is a matter of the greatest importance, as the success of the whole operation depends on this; doubtless many of the unfavorable opinions which have been expressed respecting percolation have their origin in the faulty formation of the percolator. Many forms have been suggested, varying from a narrow cylinder up to an almost funnel-shaped truncated cone; as a matter of fact no one form is suitable in all cases. A drug which swells greatly when soaked requires a less cylindrical percolator; and the larger the quantity operated upon, as a rule, the wider the percolator may be in proportion to its length. It is evident that the taller the column of drug within the percolator the more thoroughly saturated will the menstruum be as it flows from the apparatus, provided, of course, that other circumstances are the same. But this principle can be carried too far, for the liquid might become so "syrupy" that the progress of the operation would be intensely tedious. It may be taken as a rule that the length of a percolator for pharmaceutical purposes should not exceed five times nor be less than one and a half times its greatest diameter: a small percolator of less than 8 fl. ounces capacity may be of the former dimensions if used for compact drugs; large ones holding four gallons or upwards may approach the latter proportions. Fig. 63 will illustrate the general form of the body of a percolator. It consists of a cone which tapers uniformly for nearly the whole of its length, then suddenly contracts, forming a shoulder and a neck below. The smaller diameter (C D) at the shoulder should not be more than three fourths or less than one half that at A B, which bears the relation above mentioned to the length E F.

It has frequently been stated that a cylinder or an almost cylindrical cone is the best form of percolator, but there is one most important objection to that form, viz. whilst the drug is being completely soaked with menstruum it swells, and in a cylinder this swelling will cause a great pressure in a direction perpendicular to the side of the vessel, and so tend to block or even break the percolator; if, however, the form be such as above stated, this pressure is not in a direction perpendicular to the side, consequently the drug can rise to a small extent, and thus avoid a "block."

Regulation of Speed.—The rate at which percolation proceeds is another matter which requires careful attention, and for regulating this various plans are adopted. It is evident that unless some special provision be made the rate will depend mainly upon the resistance offered by the drug itself, which will vary at different stages of the operation. Perhaps the commonest method is to have the neck somewhat elongated, and closed by a tap which can be turned off or on at will. It is, however, but imperfect and difficult to clean, and for glass and earthenware, expensive. A better plan is that which is official in the U. S. P. The neck is closed by a perforated cork bearing a short glass tube, H (Fig. 63), projecting an inch or so below the cork, but not projecting within the percolator; to this tube is attached an india-rubber tube (I J) rather longer than the percolator itself, and having a second small glass tube (K) inserted in the open end. By raising or lowering this rubber tube the flow of liquid can be regulated or entirely stopped: if the open end of K be raised to a higher level than the liquid in the percolator, the flow ceases; if it be lower, the rate of flow is proportional to the difference between the height of the two. The percolator is supported on a suitable stand, and a bottle or other suitable vessel is used to receive the *percolate.* This method presents the disadvantage of the use of rubber, which with strong spirituous menstrua becomes sticky and to a small extent dissolved.

Fig. 63.

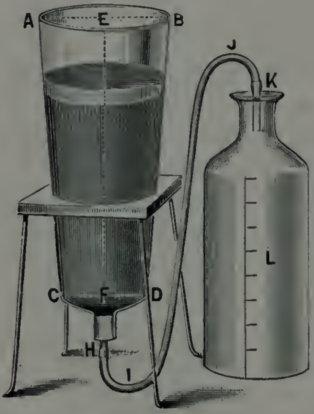
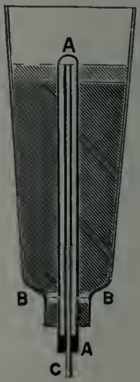


Fig. 64.



A third plan is by the use of a double tube arrangement, as shown in Fig. 64. The wide outer tube (A A) has two small holes (B B) just above the cork, and is itself closed by a cork carrying the narrow tube C, open at both ends. This narrow tube slides freely up and down, so that its upper end can be raised above the liquid which rises in the outer tube (and consequently above that in the body of percolator), the whole being then at rest; or it can be depressed to any required point to increase the rate of flow.

This alone will not ensure a *uniform* rate, which is desirable, and can be best secured by keeping the level of the liquid above the drug at a constant point. The simplest way to arrange this is by inverting a bottle containing the menstruum, and supporting it so that its open neck is about half an inch above the surface of the powder; as fast as liquid flows from the percolator more will thus be supplied. The speed to be aimed at depends upon the quantity operated upon, the nature of the preparation required, as well as the nature of the drug. It is clear that for weak preparations like the majority of tinctures a greater speed is allowable than for strong ones like fluid extracts, in which the greatest economy of menstruum is especially to be observed. Except when very large quantities are being made the percolate should always come in divided drops for fluid extracts, and in the thinnest possible column for tinctures.

Fineness of Powder.—We now come to the question of the fineness of the drug. First and foremost in this matter is uniformity; for percolation it is *essential* that the drug shall not be partly in pieces like peas and partly in dust. A No. 40 powder, *e. g.*, should almost entirely pass through a No. 40 sieve, and nearly all should be retained by a No. 60. It is evident that if large pieces be present they will encourage the formation of channels around them, and will not be properly permeated; whilst the presence of dust will tend to clog the percolator by offering greater resistance to the flow of liquid. The same general remarks apply as for maceration: fibrous or hard woody or horny drugs such as cinchona and nux vomica, must be in finer powder (No. 80 or 60); whilst spongy drugs consisting mostly of parenchymatous tissue (*e. g.* orange and buchu) are better in coarse powder (No. 20 to No. 12).

Packing and Percolating.—To obtain a satisfactory result from percolation the "packing" must be carefully done, and the proper degree of pressure can only be judged as the result of practice.

The general method of packing is as follows:—The drug, having been reduced to the required degree of powder, is thoroughly stirred up with about one-third to two-thirds its weight of the menstruum in a suitable vessel (ordinary earthenware jar as used for ointments, &c.) till uniformly mixed, then covered up, and allowed to stand about six hours or over night to swell. At the expiration of this time the mixture should be rubbed through a coarse sieve to break down any aggregations of particles, and having covered the aperture of the percolator with a loose plug of cotton wool the damp powder is introduced in small quantities at a time, pressing each lot down by means of a small block of hard wood attached to a suitable handle. The degree of pressure will vary with the drug; woody and hard drugs should be pressed *firmly*, whilst such drugs as orange or rhubarb should receive only just sufficient pressure to render the whole uniformly close. When the whole has been introduced, a disc of filter-paper, deeply cut around the edges, is placed on the surface with the object of distributing the menstruum evenly over the surface, and the cover of the percolator put on, with the bottle for automatic supply of menstruum. The tap of the percolator being open, the liquid gradually permeates the mass, and should descend regularly at all parts. This can readily be observed if a glass percolator be used; if it descends more rapidly in some parts than others it is evidence of careless packing or unequal comminution. As soon as the cotton wool is soaked the tap is turned off, or the tube raised so that no percolate can escape, and the whole is left to macerate for two days, after which percolation is allowed to procced, and carefully regulated until the proper speed is secured. This may continue until the required volume of percolate is obtained.

Official Percolation.—Percolation as above described is only officially employed for the liquid extracts of hamamelis and hydrastis, and slightly modified for tinctures of hamamelis, hydrastis, and strophanthus; but it might with advantage be extended to the great majority of B. P. preparations, which are now made by the methods of percolation and maceration combined.

Sometimes it is better to pack the powder in a dry state, and add the menstruum afterwards; this method should be adopted when very volatile solvents, such as ether or chloroform, are employed, and might also be used with spirit in those cases in which the drug does not swell to any considerable extent. This method is officially employed for oleo-resin of cubebs, for removing oil from strophanthus and stramonium previously to making the tincture and extract, and for strong tincture of ginger and syrup of rhubarb. It is certainly unnecessary for these last two, but might well be extended to blistering liquid and ethereal tincture of lobelia.

The combined method of macero-percolation official in the B. P. will be described under the section devoted to tinctures; it is used, with certain modifications as to proportions, in many other preparations, *e. g.* extracts, fluid extracts, syrups, &c.

Recovery of Menstruum.—The above description of percolation indicates no provision for recovery of the menstruum which remains soaked up in the marc. This may be done in three ways: 1st, by pressing the marc, as when maceration is employed; 2nd, by introducing the damp marc into a still, together with enough water to just cover the whole, and distilling; and 3rd, by displacement with water. In most cases this last is the most satisfactory, although it is frequently inappropriate on account of the great amount of swelling which occurs; this is the case with orange, senna, and gentian, the percolation being almost completely stopped. The disadvantage of water is that, being heavier than the spirit used, diffusion is thereby encouraged, and consequently the last portions of spirit which percolate will be weakened and loaded with mucilaginous matter. The author's experience, however, is distinctly in favour of this method, for by its means the most complete recovery of spirit can be accomplished.

The best plan is to use about 2 to 4 fl. ounces of menstruum for every pound of drug in addition to the actual quantity of percolate required, and then the preparation will be complete before water passes through; the remaining small quantity of spirit may be recovered from the weak percolate which follows, by distillation. It is also advantageous to use camphor or chloroform water in place of water for the displacement, thereby retarding the fermentation which will occur with many drugs.

Upward Displacement.—Mr. W. Elborne (‘Pharm. Journ.’ [3], x, p. 973) has suggested a method of upward percolation and displacement in which the spirit is displaced by means of water passing up from below; he states that the recovery of spirit is more complete: but the method does not appear to have been largely used, probably because the whole of the spirit must be added to the drug at once, or upward percolation would be unsatisfactory, on account of the first and stronger percolates tending to diffuse into the fresh spirit from below by the action of gravity.

Hot Percolation.—It is frequently advantageous to subject the contents of a percolator to the action of a solvent at a temperature approaching its boiling-point. This may be accomplished by having the percolator surrounded by a water or steam jacket; in this case the top of the percolator should communicate with a small upright condenser, to avoid loss of solvent.

The number of preparations for which some variety of percolation is used is very large; they will now be considered in classes.

Questions on Chapter XVII.

1. What is percolation? What is the form of percolator best suited for an operation employing about one pound of drug?
2. Describe carefully the operation of packing a percolator.
3. Give short notes on the comminution of drugs for percolation.
4. Give three methods of recovery of the menstruum used in percolation.
5. What is the advantage of *upward* displacement?

CHAPTER XVIII

EXTRACTION OF DRUGS (*continued*).

Tinctures.

Methods of Preparation.—The official tinctures are prepared by five methods: (1) simple admixture of liquids; (2) simple solution of solids; (3) maceration; (4) percolation; (5) maceration and percolation combined.

Those prepared by the first two methods have already been noticed; they are, tincture of tolu (p. 69), acetate and perchloride of iron (p. 61), compound tincture of chloroform (p. 60), chloroform and morphine (p. 82), nux vomica (p. 69), podophyllin (p. 69), quinine (p. 69), ammoniated quinine (p. 95), iodine (p. 69), and Indian hemp (p. 69).

The process of maceration is commonly used in the following cases:

(*a*) Drugs like aloes, catechu, or asafœtida, which are mostly soluble in the menstruum, or are of a very resinous nature, and consequently liable to run together in the percolator; an official exception is myrrh, which, however, is just as well prepared by maceration.

(*b*) Drugs which do not admit of a degree of comminution suitable for percolation, such, *e. g.*, as fresh orange and lemon peel.

(*c*) When the proportion of menstruum is very large, *e. g.* compound tincture of camphor, cantharides, quassia, and lavender, when no advantage would attend percolation.

(*d*) When volatile solvents are employed, which would require special precautions to avoid loss by percolation: ethereal tincture of lobelia, ammoniated tincture of valerian.

The student should now prepare at least two of the official tinctures by maceration. The general directions are indicated by the following example:

"Take of—

Catechu, in coarse powder . . .	2½ ounces.
Cinnamon bark, bruised . . .	1 ounce.
Proof spirit	1 pint.

"Macerate for seven days in a closed vessel, with occasional agitation; strain, press, filter, and add sufficient proof spirit to make one pint."—B. P.

In the case of very soluble drugs—for example, aloes and asafœtida—it is necessary to modify this by using only three fourths of the spirit for the maceration, and making up to volume after filtration, on account of the fact that the liquid increases in volume by solution of so large a quantity of solid matter.

The general remarks given on pp. 182–3 apply equally to the preparation of tinctures by maceration.

The Menstruum.—The official menstruum employed is usually either proof spirit or rectified spirit; the exceptions are noted in the tables following. There is, however, much room for improvement in the selection of menstrua; in many cases spirit of an intermediate strength is more suitable, and for some tinctures a mixture of equal parts of rectified spirit and water is preferable to proof spirit. Messrs Wright and Farr have contributed a valuable series of notes on this subject (see 'P. J.' [3], vols. xxi and xxii, and 'C. and D.,' 1891–2); they show that for aconite, cinchona, conium, and veratrum, a spirit containing 70 per cent. of alcohol by volume is the most suitable strength, whilst for colchicum and jaborandi 50 per cent. of alcohol is the best.

Table of Tinctures prepared by Maceration (all for seven days).

Name of tincture.	Part used.	Menstruum.	Strength.	Note.
Tinctura Lavandulæ Composita	Volatile oil	Rectified spirit	1 in 213⅓ by measure, with ⅑ oil of rosemary, 1⅔ each cinnamon and nutmeg, and 3⅓ red sandal-wood	
„ Benzoini Composita	Balsam (gum-resin), in coarse powder	„	Benzoin 1, with styrax ¾, tolu ¼, Socot. aloes $\frac{3}{11}$ in 10	1
„ Asafœtidæ .	Gum-resin (small fragments)	„	1 in 8	2

Name of tincture.	Part used.	Menstruum.	Strength.	Note.
Tinctura Aurantii recentis	Fresh peel	Rectified spirit	1 in 3⅓	1
,, Camphoræ Composita	Camphor, opium in powder	Proof spirit	Opium 1, with benzoic acid 1, camphor ¾, and oil of anise ¾ (by measure), in 220	
,, Cantharidis	Coarsely powdered flies	,,	1 in 80	3
,, Aloës . .	Socotrine aloes coarsely powdered	,,	Aloes 1, and extract of liquorice 3 in 40	1
,, Quassiæ .	Rasped wood	,,	1 in 26⅔	
,, Opii . .	Powdered drug	,,	1 in 13⅓	4
,, Aurantii .	Bruised peel	,,	1 in 10	
,, Catechu .	Coarse powder	,,	1 in 8, with cinnamon bark ⅖	
,, Cocci . .	Powdered insects	,,	1 in 8	
,, Limonis .	Fresh peel	,,	,,	1 & 5
,, Lobeliæ Ætherea	Powdered herb	Spirit of ether	,,	
,, Valerianæ Ammoniata	Rhizome and rootlets in No. 40 powder	Aromatic spirit of ammonia	,,	
,, Guaiaci Ammoniata	Resin in powder	,,	1 in 5	1
,, Opii Ammoniata	Powdered opium	Rectified spirit 4, strong solution of ammonia 1	Opium 1, with saffron and benzoic acid, of each 1⅘, and oil of anise ⅗ (fluid) in 87½	
,, Kino . .	Coarse powder	Rectified spirit 12, water 5, glycerine 3, by measure	1 in 10 of mixture	1 & 6

Note 1.—The whole of the spirit should not be added at first, as this tincture commonly gains volume.

Note 2.—Asafœtida must be carefully selected; few drugs are more largely adulterated: stones, chalk, and mortar are the usual adulterants.

Note 3.—This is a very unsatisfactory preparation, the active principle being so little soluble in proof spirit that the tincture is a saturated solution; a mixture of rectified spirit with 10 per cent. of acetic ether would be preferable.

Note 4.—It is far better to use moist opium, the aroma being more perfectly retained and the morphine more completely extracted. The opium should be sliced, and macerated in the correct quantity of distilled water, after allowing for that natural to the drug,* until quite soft, which will be in a day or two; the rectified spirit then added, and the whole allowed to macerate for three days, with occasional agitation; then decanted, pressed, filtered, and made up to measure with proof spirit. Moist opium of good quality contains as much morphine as the official powdered opium, but it should always be assayed (see Chap. XXIII).

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* Moist opium contains about 20 per cent. of water.

Note 5.—A far better preparation could be made like the corresponding tincture of fresh orange peel, or with a mixture of S. V. R. 3 fl. parts, water 1 fl. part.

Note 6.—The glycerine is added with the object of preventing gelatinisation, which otherwise occurs.

Percolation has been already described. The B. P. directs the following to be prepared by percolation; the student should prepare those marked *.

Tinctures prepared by Percolation.

Name of tincture.	Part used.	Menstruum.	Strength.	Note.
Tinctura Hamamelidis	Bark in No. 20 powder	Proof spirit	1 in 10	
* ,, Hydrastis	Rhizome and rootlets in No. 60 powder	,,	,,	
* ,, Strophanthi	Seeds in No. 30 powder	Rectified spirit	1 in 20	1 and 2
,, Zingiberis fortior	Rhizome in fine powder	,,	1 in 2	2

The directions for percolation given in the B. P. differ somewhat from those given above; the powder is allowed to stand twenty-four hours with sufficient menstruum to moisten it, and percolation is then proceeded with immediately after packing.

Note 1.—The strophanthus, in No. 30 powder, is dried at 110° F. (43·3° C.) and packed dry, the fixed oil removed by percolating with ether free from alcohol or water, in which the oil dissolves but the bitter principle remains insoluble; if the ether were not pure it would dissolve some of the bitter. The marc is removed from the percolator, and dried at or below 120° F. (48·9° C.), again rubbed to powder, re-packed in percolator moistened with spirit, allowed to stand forty-eight hours, and exhausted by percolation with rectified spirit to produce 10 fl. parts. It is then made up to 20 fl. parts by addition of more rectified spirit.

Note 2.—These are packed dry, without previous moistening.

Macero-percolation.—The official method of preparing the majority of the tinctures is by a combination of maceration and percolation, as in the following example, which should be prepared by the student.

Tinctura Digitalis.

"Take of—

Foxglove leaves, in No. 20 powder	$2\frac{1}{2}$ ounces.
Proof spirit	1 pint.

"Macerate the foxglove for forty-eight hours in 15 fl. ounces of the spirit in a closed vessel, agitating occasionally; then transfer to a percolator, and when the fluid ceases to pass continue the percolation with the remaining 5 ounces of spirit. Afterwards subject the contents of the percolator to pressure, filter the product, mix the liquids, and add sufficient proof spirit to make one pint."

The maceration may be conducted as usual, and at the end of the specified time the whole contents of the bottle or jar are bodily turned into a long and narrow percolator, allowed to stand a few hours for the contents to pack themselves by the action of gravity, and percolation then carried out as directed; or the greater part of the liquid may be decanted, and the drug with adhering menstruum packed in the percolator. Either of these methods is, however, clumsy, and it is preferable to carry out the maceration in the percolator itself; better still, however, is to prepare tinctures by the method of percolation alone. Messrs Wright and Farr, in the series of notes above referred to, have clearly shown the advantage of percolation over methods of maceration or this official method in almost all the tinctures examined. Percolation might be expected to prove the most advantageous, for there is always a certain amount of liquid retained by the drug, even after the most careful pressure, which we are directed to make up by adding more menstruum. In the case of maceration this liquid is clearly as strong, and probably stronger than the decanted tincture; but by percolation the last portions are almost inert, the great bulk of the soluble matter being removed from the drug by the first few ounces (on pint quantities) of menstruum: by the B. P. method a residual liquor of intermediate value is retained.

Tinctures officially prepared by Macero-percolation with Rectified Spirit.

Name of tincture.	Part used.	Degree of powder.	Strength.	Note.
Tinctura Capsici	Fruit	Bruised	1 in $26\frac{2}{3}$	
,, Arnicæ	Rhizome and rootlets	No. 40	1 in 20	
,, Aconiti	Root	,,	1 in 8	
,, Cinnamomi	Bark	Coarse powder	,,	1
,, Cubebæ	Fruit	In powder	,,	1
,, Laricis	Bark	No. 40	,,	
,, Myrrhæ	Gum-resin	Coarse powder	,,	2
,, Sumbul	Root	No. 40	,,	
,, Zingiberis	Rhizome	In powder	,,	1
,, Pyrethri	Root	No. 40	1 in 5	
,, Veratri Viridis	Rhizome and rootlets	,,	,,	

It should be noted that *all* the rectified spirit tinctures prepared by this method are *simple* tinctures (*i. e.* contain only one drug).

Note 1.—It would have been preferable to have ordered these drugs to be in No. 40 powder, as more definite.

Note 2.—No. 16 powder is about the most desirable.

Tinctures officially prepared by Macero-percolation with Proof Spirit.

Name of tincture.	Part used.	Degree of powder.	Strength.	Note.
Tinctura Cardamomi Composita	Seeds	Bruised	1 in 80, with caraway 1, cinnamon 2, cochineal $\frac{1}{2}$, raisins 8	1
,, Belladonnæ	Leaves	No. 20	1 in 20	
,, Croci	Stigmas	Whole	,,	
,, Gentianæ Composita	Root	Cut small and bruised	1 in $13\frac{1}{3}$, with orange peel $\frac{1}{2}$ and cardamoms $\frac{1}{6}$	2
,, Cinchonæ Composita	Bark	No. 40	1 in 10, with orange peel $\frac{1}{2}$, serpentary $\frac{1}{4}$, saffron $\frac{1}{16}$, cochineal $\frac{1}{32}$	
,, Rhei	Root	No. 20	1 in 10, with cardamoms, coriander, and saffron, of each $\frac{1}{8}$	
,, Buchu	Leaves	,,	1 in 8	
,, Calumbæ	Root	Cut small	,,	2
,, Cascarillæ	Bark	No. 40	,,	
,, Chiratæ	Herb	Cut small and bruised	,,	2
,, Cimicifugæ	Rhizome and rootlets	No. 40	,,	
,, Colchici	Seeds	Finely comminuted	,,	3

Name of tincture.	Part used.	Degree of powder.	Strength.	Note.
Tinctura Conii . .	Fruit	Finely comminuted	1 in 8	
,, Digitalis .	Leaves	No. 20	,,	
,, Gallæ . .	Galls	No. 40	,,	
,, Gelsemii .	Root	,,	,,	
,, Hyoscyami .	Leaves or flowering tops	No. 20	,,	4
,, Jalapæ .	Tubers	No. 40	,,	5
,, Krameriæ .	Root	,,	,,	
,, Lobeliæ .	Herb	,,	,,	
,, Lupuli .	Inflorescence	Whole	,,	6
,, Sabinæ .	Tops	Coarse powder	,,	
,, Scillæ . .	Bulb	Bruised	,,	1
,, Senegæ .	Root	No. 40	,,	
,, Serpentariæ .	Rhizome and rootlets	,,	,,	
,, Stramonii .	Seeds	Bruised	,,	7
,, Valerianæ .	Rhizome and rootlets	No. 40	,,	
,, Sennæ . .	Leaves	Broken small	1 in 8, with caraway $\frac{1}{5}$, coriander $\frac{1}{5}$, and raisins $\frac{4}{5}$	1 & 8
,, Cinchonæ .	Bark	No. 40	1 in 5	9
,, Ergotæ .	Mycelium	Finely comminuted	1 in 4	10
,, Jaborandi .	Leaves	No. 40	,,	

Note that all the tinctures of the strength 1 in 8, with the single exception of senna, are simple tinctures.

Note 1.—Owing to the nature of the drugs, these tinctures are better prepared by the B. P. method or by maceration than by percolation alone, on account of the difficulty of packing.

Note 2.—The drugs are ordered "cut small;" any method of percolation is inappropriate to such a degree of division; maceration for seven days would be better, or better still to macerate with two-thirds of the spirit, then press out, and again macerate with the remaining third of the spirit, finally pressing again, filtering and adding spirit to measure. In the case of gentian the drugs might well be reduced to No. 16 powder, and the tincture prepared by percolation.

Note 3.—The seeds are rightly ordered "finely comminuted," as the drug is so "horny" that it is but imperfectly exhausted when in coarse powder; a No. 60 or No. 80 powder is appropriate ('P. J.' [3], xxii, p. 364).

Note 4.—This as well as the other preparations of henbane is ordered to be made from the biennial plant; Gerrard has shown that this possesses very little if any advantage over the annual variety (see 'P. J.' [3], xxi, p. 212).

Note 5.—Jalap being of a resinous nature, it would be preferable to use a stronger spirit (3 parts S. V. R. to 1 of water).

Note 6.—A stronger tincture is obtained by using the hop in No. 20 powder.

Note 7.—Wright and Farr have shown that a tincture prepared with the bruised seeds is equally as strong as with a fine powder, and not so loaded with oil.

Note 8.—B. S. Proctor has shown that the purgative principle of senna is only slightly soluble in proof spirit, consequently the preparation is unsatisfactory. A better tincture could be made with spirit consisting of rectified spirit 2 fl. parts, water 3 fl. parts.

Note 9.—Although the cinchona is officially ordered to contain from 5 to 6 per cent. of alkaloids, this tincture is not of even approximately uniform strength; the whole of the alkaloids are not extracted, and the amount actually obtained varies much according to the nature of the particular bark used.

Note 10.—It is unnecessary to reduce the ergot to fine powder; a No. 20 powder is quite fine enough. A mixture of rectified spirit 1 part, water 1 part, would be a better solvent, and extract less of the fatty oil.

Questions on Chapter XVIII.

1. By what methods are tinctures prepared? Give three examples of each.
2. Describe minutely the preparation of tinctures of hamamelis and orange.
3. In what cases should maceration be adopted, and why?
4. Give reasons for the following:—Use of ether in preparation of tincture of strophanthus, glycerine for kino.
5. Describe the B. P. process of macero-percolation, using compound tincture of cinchona as an example.
6. Comment upon official formulæ or directions for tinctures of benzoin, aloes, opium, lemon, stronger ginger, cubebs, compound cardamoms, calumba, colchicum, jalap, hop, gentian, and ergot.
7. Why is tincture of senna unsatisfactory? In what way may it be improved?

CHAPTER XIX

EXTRACTION OF DRUGS (*continued*)

Liquid Extracts—Extracts

Strength of Liquid Extracts.—In the majority of cases it has become the common practice to make liquid extracts of such a strength that one fluid part shall contain the soluble matter of one part by weight of the original drug; in fact, so general has this practice become, that to avoid confusion it should always be followed out unless there is some special reason for the contrary. With the exception of liquid extracts of opium, liquorice, and fern, the formulæ of the official liquid extracts have been framed to meet this practice.

It will, therefore, be readily seen that for their production a simple process of percolation, such as has been given for tinctures, would be most imperfect, whilst ordinary maceration would be even more useless. Various methods have been devised in order to attain this degree of concentration, viz.:

Preparation.—1. The drug is exhausted with an aqueous liquid by maceration or percolation or both, the liquid evaporated to rather less than the required volume, and rectified spirit added as a preservative, the quantity needed varying with the nature of the drug. Examples:—Cascara sagrada, liquorice.

2. The drug, in powder, is macerated with a small quantity of spirit for a certain period, then pressed firmly, and the expressed liquid reserved; the marc treated with water, and again macerated and pressed. This second liquid is evaporated to a suitable degree, and then mixed with the spirituous liquid, allowed to stand, and cleared by filtration or decantation. Examples:—Sarsaparilla, dandelion.

3. An extract is prepared by any of the methods to be presently described, and dissolved in a suitable solvent. Examples :—Opium, pareira.

4. The method official in the United States Pharmacopœia, and adopted in a few instances in the B. P., is as follows :—The drug is damped as usual with a spirituous menstruum, packed in a narrow percolator, and after maceration for forty-eight hours, percolation is started; when the percolate amounts to about 80 to 85 per cent. of the desired quantity of finished preparation the receiver is changed and percolation continued to exhaustion, that is until the characteristic principle of the drug is only present in traces. This second percolate is then put into a still, the spirit recovered by distillation, the residue in still evaporated to a soft extract, which is added to the first percolate, well shaken together, and when completely dissolved or disseminated through the liquid it is allowed to stand twenty-four hours, the clear liquor syphoned off, the "bottoms" filtered, and sufficient of the distilled spirit passed through the filter to make the whole measure the required volume. Examples :—Hamamelis, hydrastis.

5. *By repercolation or fractional percolation.*—This method is not officially recognised, but it is undoubtedly by far the best for preparing the majority of the liquid extracts, and is carried out as follows :

Supposing it is required to make 40 fl. oz. of liquid extract of cimicifuga; the drug is first reduced to a uniform degree of comminution; 8 oz. of the powdered drug are then damped with rectified spirit (4 or 5 fl. oz. will be required), and mixed uniformly by thoroughly stirring together in a jar; the mixture is covered to prevent evaporation, and set aside during a night so that the drug may swell; afterwards rub through a coarse sieve to break up any aggregations of drug which may have formed, and pack in a percolator. (The double-tube percolator is admirably adapted for this purpose.) Pour on sufficient rectified spirit to saturate the drug and leave a shallow layer above the powder, then arrange for an automatic supply, and proceed as usual, macerating forty-eight hours; then start percolation. As soon as 4 fl. oz. of percolate have collected change the receiver, reserving the 4 fl. oz. Now continue percolation until sufficient has been obtained to damp 8 oz. more of the cimicifuga, which is packed in a percolator as before. The percolation in No. 1 percolator is continued till exhausted, the percolate being collected in fractions of about 8 fl. oz. each and transferred successively to No. 2. Percolation of No. 2 is continued till 8 fl. oz. are obtained, which quantity is added to the already reserved 4 fl. oz. from No. 1, after which the next 4 fl. oz. from No. 2 are used to damp powder for the third percolator. The drug in the second percolator is percolated to exhaustion just like No. 1, using the successive percolates from No. 1, and that in No. 3 is treated like No. 2, and so on until five separate percolations of 8 oz. of the drug have been conducted. From the fifth percolator the first 12 fl. oz. are added to the reserved portion, when the total bulk reserved will amount to 40 fl. oz.

The recovery of the absorbed menstruum is carried out as described before (p. 199), preferably by displacement with water, the weak liquid being reserved as menstruum for a succeeding batch of fluid extract.

Advantages of Methods.—We will now review the advantages and disadvantages of these methods. Many of the active principles of plants are greatly injured by a long-continued high temperature, consequently it must be taken as a rule in pharmacy that *drugs should not be subjected to heat unless absolutely necessary, or to avoid some more pressing disadvantage attending preparations made without heat.* Even if the active principles are not thus susceptible, the evaporation of solutions of the ordinary plant principles is attended by a very considerable darkening in colour, and on this account alone heat should be avoided if possible. For this reason processes 1 and 3 above described are to be deprecated, unless the evaporation be carried out *in vacuo,* a method which is seldom possible on the small scale; the same remark applies in a lesser degree to process 2, and still less to process 4. This last method is a good one in very many cases, as there is seldom more than one-fourth of the soluble matter present in that portion of the liquid which is subjected to heat. By repercolation no heat whatever is needed. There is, however, one minor disadvantage attending processes 4 and 5; that is, in many cases it is necessary to employ rather a stronger spirituous menstruum, thus increasing the cost: *e. g.* liquid extract of liquorice is officially ordered to be prepared by exhausting the drug with cold water, straining the liquid and raising to 100° C., filtering, then evaporating by a water-bath to a specific gravity of 1·160; to this liquid is added one sixth of its volume of rectified spirit, allowed to stand eighteen hours, and filtered; the heating causes coagulation of albumen, which is removed by the filtration through flannel, and the finished preparation keeps well. If, however, it were attempted to prepare this liquid extract by percolation with a mixture of 1 part S. V. R. and 6 parts water, the resulting preparation would contain the albumen or a considerable porportion of it, which would render it less permanent, albumen being especially liable to decomposition. Such a result need not be feared when the amount of rectified spirit reaches 25 per cent. of the finished preparation; in fact, when such is the case, liquid extracts made without heat keep far better than those made with heat, as the latter commonly deposit slowly such substances as are sparingly soluble in the mixture of spirit and water, though soluble in water alone or spirit alone; liquid extract of cascara may be cited. This is only what might be expected; for, by method 1, the watery liquid will extract from drugs much mucilaginous, albuminous, saccharine, or even starchy matter, this being precipitated more or less completely on addition of the spirit, but not all within the twenty-four hours or so which is commonly allowed before filtration. By process 2 the additional disadvantage of the precipitation of resinous matter from the spirituous liquid is also incurred. However, by all methods changes in temperature will usually cause slight deposits to occur in liquid extracts (least of all in those prepared by repercolation), as one or other of the plant principles commonly exists in the product to the point of saturation; this is especially the case if the extracts have been prepared during warm weather.

Plant Principles.—The following list indicates the general behaviour of the commonly occurring plant principles towards various solvents, and will serve as a guide to the student; it will also show how important it is that he should gain an insight into the nature of the constituents of each drug which he has to manipulate.*

Constituent.	Ether.	Rectified spirit.	Proof spirit.	Cold water.	Boiling water.
Fixed oils	Freely soluble	Slightly soluble	Almost insoluble	Insoluble	Insoluble.
Volatile oils	Freely soluble	Freely soluble	Slightly soluble	Nearly insoluble	Nearly insoluble.
Chlorophyll	Soluble	Soluble	Slightly soluble	Insoluble	Insoluble.
Waxes	Soluble	Slightly soluble	Nearly insoluble	Insoluble	Insoluble.
Resins	Variable	Freely soluble	Moderately soluble	Insoluble	Nearly insoluble.
Glucosides	Variable	Mostly soluble	Mostly soluble	Many soluble	Many soluble, but some decomposed.
Alkaloids (free)	Variable	Soluble	Soluble	Generally insoluble	Insoluble or nearly so.
Alkaloidal salts	Generally insoluble	Generally soluble	Soluble	Soluble	Soluble.
Tannins	Generally insoluble	Soluble	Soluble	Generally soluble	Generally soluble, some decomposed.
Gum, mucilage	Insoluble	Insoluble	Nearly insoluble	Soluble	Soluble.
Dextrine	Insoluble	Insoluble	Slightly soluble	Soluble	Soluble.
Sugar (dextrose)	Nearly insoluble	Soluble	Soluble	Soluble	Soluble.
Sugar (saccharose)	Insoluble	Slightly soluble	Slightly soluble	Soluble	Soluble.
Albuminoids	Insoluble	Insoluble	Insoluble	Soluble	Mostly insoluble.
Starch	Insoluble	Insoluble	Insoluble	Insoluble	Soluble.
Cellulose	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble.

Menstrua for Liquid Extracts, and Extracts.—The menstrua employed for extraction are commonly either hot or cold water, and spirit of varying strength, although in one official case ether is used (*Extractum Filicis Liquidum*). A glance at the above table will indicate how widely different in constitution the various liquids so obtained must necessarily be.

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* It must be understood that there are exceptions which are not provided for in this table, *e. g.* castor oil is freely soluble in rectified spirit.

The student should now prepare the following liquid extracts:

Extractum Ergotæ Liquidum.

"Take of—

Ergot, crushed	8 parts.
Cold distilled water	60 ,,
Rectified spirit	3 fl. ,,

"Digest the ergot in forty parts of the water for twelve hours. Draw off the infusion and repeat the digestion with the remainder of the water. Press out, strain, and evaporate the liquors by the heat of a water-bath to five and a half fl. parts; when cold, add the spirit. Allow it to stand for an hour to coagulate, then filter. The product should measure eight fl. parts."—B. P.

Cold water exhausts ergot equally as well as hot; moreover the latter liberates the oil from the drug, which, floating on the surface, impedes evaporation. The first straining may be through muslin or flannel; the weaker liquor should in this and in all such cases be evaporated first, so as to avoid subjecting the stronger solution to any unnecessary heat. The addition of spirit coagulates the mucilage, which, together with albuminous compounds previously coagulated by the heat of evaporation, is removed by the final filtration. The B. P. formula allows half a fl. part for waste absorbed by the magma upon the filter. *One* hour is scarcely long enough for satisfactory precipitation of the mucilage, especially as the admixture of spirit causes a rise of temperature.

"*Extractum Cascaræ Sagradæ Liquidum.*

"Take of—

Cascara Sagrada, in coarse powder	4 parts.
Rectified spirit	1 fl. part.
Distilled water	a sufficiency.

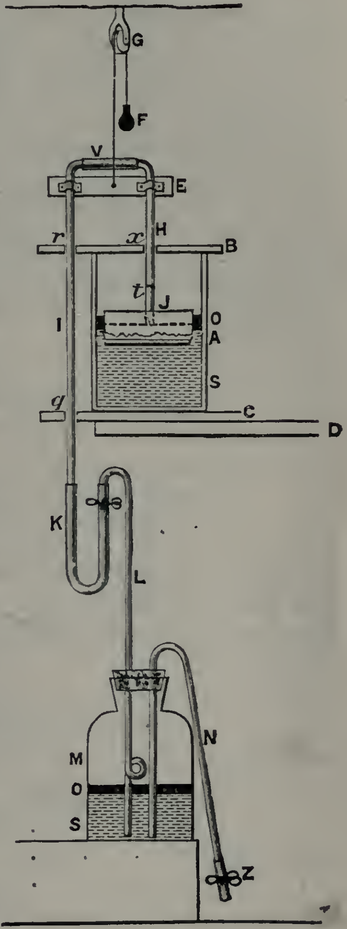
"Boil the bark in three or four successive quantities of the water until exhausted. Evaporate the strained liquors by a water-bath to three fl. parts; when cold, add the spirit; allow the mixture to stand some hours, then filter and make up the volume to four fl. parts with distilled water."—B. P.

Liquid extract of Rhamnus frangula is prepared exactly like the above.

The barks are rightly ordered in *coarse* powder; in fact, it would be better to use them simply bruised; when powdered, the boiling water seems to extract much more mucilaginous matter, which further impedes the necessarily slow filtration. Enough water should be used for each boil to just cover the bark.

Upward Filtration.—The final filtration, which is very tedious, is best applied as follows:—Allow the mixture to stand in a cool place for a week, then syphon off the clear liquid and reserve the thick portion. After several batches have been made, the bottoms of all are mixed and subjected to upward filtration, which is often advantageously applied to such slimy deposits, avoiding as it does the clogging of the pores of the filter. This is best accomplished by means of Warner's upward filter (Fig. 36), or of the apparatus described by F. C. J. Bird in the 'Pharmaceutical Journal' (3), xviii, p. 703 (Fig. 65). A is a stoneware jar, of about 2 gallons capacity, placed on a shelf at a height of five or six feet above the vessel M. It is secured to a board, C, of suitable dimensions, perforated by a circular hole, *g*. B is of wood, 3 inches in width, and also perforated by two holes *x* and *r*. C, A, and B are securely fastened together by string or other suitable means. J is the filter proper, and consists of a circular box, closed at the top and open at the bottom, and about $\frac{3}{4}$ inch less in diameter than A. J is divided at the centre by a partition, which thus forms an air-tight chamber in the upper portion. The tube *t* passes through this chamber, and communicates with the lower half of J, its upper end being connected to the glass tube H by rubber tubing. Over the mouth of J is stretched the filtering medium, consisting of three layers, calico, paper, and flannel, the last being on the outside. E is a bar of wood, to which the glass tubes H and I are firmly attached, J, H, I, E forming a rigid system, partially counterbalanced at its centre of gravity by the weight F, through the cord and pulley G, the whole being capable of free motion up and down, so that J rises and falls with the liquid in the interior of A. The tubes H and I work through the holes *x*, *r*, and *g*, which serve as guides. The weight F

Fig. 65.



should be such that when A contains no liquid, J just descends freely to the bottom of the jar. K is a piece of india-rubber tubing connecting I and L, so as to allow of free motion of I. L is a glass tube passing into the bottle M, and twisted once as shown near the bottom. A syphon and pinch-cock, N Z, are required to draw off the filtrate from M.

If it is desired to start filtration, A is filled with liquid, when by the buoyant action of the air chamber in J, aided by the weight F, the filter rises to the surface. A cork is inserted in the bottom of L, and the end of the rubber tube V removed from I. Through V and I, I K L and H J are filled with liquid (preferably bright). The connection at V is again made and secured, and a layer of colourless, heavy petroleum oil about ⅓ of an inch in depth poured on the two surfaces O, O, of the liquids in A and M. The object of the petroleum is to form a layer to protect the surface of the liquid from oxidation by the air; it is of course unnecessary in the case of liquids such as the one now under consideration, which are not impaired by this cause.

As soon as the end of L is unclosed filtration commences, and goes on continuously. All joints must be bound with waxed thread or wire, and thick india-rubber tubing used, to avoid collapse of its walls and consequent stoppage of the flow; the filter J should also be thoroughly soaked in melted paraffin.

"*Extractum Hamamelidis Liquidum.*

"Take of—

Hamamelis leaves, in No. 40 powder .	20 parts.
Rectified spirit, Distilled water, } of each	a sufficiency."

The powder is moistened with 8 fl. parts of a mixture of 2 fl. parts of water and 1 fl. part of spirit, packed in a percolator, and percolation carried out as described above by *method* 4.

"*Extractum Sarsæ Liquidum.*

"Take of—

Jamaica sarsaparilla, in No. 40 powder .	10 parts.
Proof spirit	10 fl. parts.
Sugar	1¼ part.
Distilled water	60 fl. parts.

"Mix the sarsaparilla with the spirit, and macerate in a closed vessel for ten days, then press out 5 fl. parts of liquor and set this aside. Mix the pressed residue with the water, and macerate at 160° F. (71·1° C.) for sixteen hours, then strain and press out the liquid, dissolve the sugar in this, and evaporate in a water-bath to about 4½ fl. parts.

Mix the two liquids and make up the volume to 10 fl. parts by the addition of distilled water."

Great pressure required to obtain 5 fl. parts of spirituous liquor; water at 71° C. is used, as it extracts the drug rapidly, but does not dissolve the starch; the previous use of spirit retains much aroma, the sugar prevents a resinoid substance from separating during the evaporation.

Liquid extract of taraxacum is made similarly, without the use of sugar.

Table of Liquid Extracts of B. P.

Name of liquid extract.	Menstruum.	Method of exhaustion.	Part used.	S. V. R. added to final product.	Remarks.
Extractum Belæ Liq. .	Cold water	Maceration, in 3 portions	Dried fruit	3 to 13	Note 1
,, Ergotæ Liq. .	,,	Maceration, in 2 portions	Mycelium	3 to 5½	P. 214
,, Glycyrrhizæ Liq.	,,	,,	Dried root	1 to 6	P. 212
,, Opii Liq. .	,,	Maceration	Extract	1 to 4	Note 2
,, Cascaræ Sagradæ Liq.	Boiling water	Decoction, several portions	Bark	1 to 3	P. 214 Note 3
,, Rhamni Frangulæ Liq.	,,	,,	,,	,,	,,
,, Pareiræ Liq. .	Rectified spirit 1, distilled water 3	Solution	Extract	—	P. 77
,, Hamamelidis Liq.	Rectified spirit 1, distilled water 2	Percolation (method 4)	Leaf	—	P. 216
,, Hydrastis Liq.	Rectified spirit 1, distilled water 1	,,	Rhizome and rootlets	—	Note 4
,, Cocæ Liq. .	Proof spirit	Macero-percolation	Leaf	—	Note 5
,, Cimicifugæ Liq.	Rectified spirit	,,	Rhizome and rootlets	—	,,
,, Taraxaci Liq.	Proof spirit, followed by cold water	Maceration, pressure, maceration	Dried root	—	Note 6
,, Sarsæ Liq. .	Proof spirit, followed by water at 160° F.	Maceration, pressure, digestion	Root	—	P. 216
,, Cinchonæ Liq.	Acidulated water and glycerine	Macero-percolation	Bark	12½ to 87½	Chap. XXIII
,, Filicis Liq. .	Ether	Percolation	Rhizome	—	Note 7

Note 1.—The preparation is cleared by decantation, being slow in filtration; there is, however, but little deposit, the quantity of spirit being only just sufficient to keep the preparation, and not enough to coagulate mucilage which is purposely retained in the final product.

Note 2.—**Pumice as a Filtering Medium.**—The extract should be rubbed smooth with a little water, the remainder of the water added, and the mixture allowed to stand for one hour, stirring occasionally; the spirit is then added, and the mixture filtered. The mixing of the spirit causes a slight rise of temperature; it is, therefore, advantageous to let the mixture stand in a very cold place for a short time before filtering; by this means the preparation is in great measure prevented from throwing out a sediment after filtration. It may be filtered through paper; the use of pumice or silica, however, assists in the production of a bright filtrate. A small quantity of the pumice or silica in about a No. 60 powder is agitated with the liquid, and before it has settled it is transferred to a paper filter, the first runnings being returned to the filter.

Note 3.—Moss has shown that the deposit which forms on keeping this fluid extract, and whilst evaporating the decoction, is not devoid of purgative action. A permanent preparation may be made by percolation with proof spirit, or by evaporating an aqueous extract till hard, then macerating in cold water, straining and evaporating, finally adding the spirit as in B. P. In this latter case the resins are rejected; see 'P. J.,' xxii, p. 250, and 'C. and D.,' i, 1892, p. 262.

Note 4.—Prepared like liquid extract of hamamelis.

Note 5.—The B. P. directions are to macerate 1 part of the drug with 2 parts of the menstruum for forty-eight hours, then transfer to a percolator; reserve $\frac{3}{4}$ fl. part of the percolate, and evaporate the remainder (when exhausted) by distillation and evaporation to a soft extract; and adding this to the reserved portion, finally adding menstruum to make 1 fl. part. By this method it is obvious that considerably more than half of the soluble matter is subjected to much heat, it is therefore *bad*, and should be replaced by percolation as for liquid extracts of hydrastis, or better still by repercolation.

Note 6.—Far better prepared by percolation (method 4).

Note 7.—**Percolation with Ether.**—The fern in *coarse* powder is packed dry and exhausted by percolation with ether. The ether is recovered by distillation and the oily extract preserved. This preparation would be more correctly classed as an oleo-resin, with Oleo-resina Cubebæ, which is similarly prepared. When ether is used as a menstruum for percolation it is necessary to have a very closely fitting cover to the percolator; the percolator should also fit the neck of the receiver accurately. The air-space above the drug communicates by a side tube with the receiver, thus permitting free flow of the percolate whilst avoiding evaporation of solvent.

Many other liquid extracts are official in other pharmacopœias, and some of these are in common use in this country.

The student should now prepare one or two liquid extracts by repercolation, so that he can observe the difference between the products by this method and the official. Liquid extracts of coca or cimicifuga, and ergot or taraxacum, will be useful practice.

Extracts.

The extracts are prepared by exhausting drugs by methods similar to those employed for liquid extracts, and evaporating to a solid or semi-solid consistence. There is also a group of eight extracts which are prepared from the juices of fresh plants, expressed as described in Chapter IX. Five of these are known as the *green extracts*, because they are made from the leaves and green portions of the plants, and are so prepared as to retain much of the natural green colour.

In preparing extracts—and the same remark applies to liquid extracts—the fresh juice or any aqueous liquid containing the soluble matter from a drug should be evaporated without delay, because these solutions are exceedingly prone to undergo fermentation ; sugar may be changed into alcohol, albuminous liquids may undergo putrefactive changes producing ammonia, and mucilaginous or saccharine substances may change into lactic or acetic acid. Each of these changes is due to the presence of certain specific forms of life which belong to the class of fungi, and are either moulds or bacilli ; the spores of these fungi are constantly present in the air, consequently no liquid capable of the changes described will be safe from deterioration under ordinary conditions. The finished extracts are not so liable to the ravages of these fungi for several reasons :—(1) As is well known, strong solutions of sugars do not undergo alcoholic fermentation ; in fact, the sugar, if in sufficient quantity, actually prevents their growth, as in syrups, treacle, jams, &c. : the extracts being nearly solid, there is no possibility of alcoholic fermentation ; the same remark applies with somewhat less force to the lactic fermentation. (2) The albumen is removed during the preparation of extracts, consequently ammonia cannot be produced as a result of putrefaction. In liquid extracts it is the alcohol which prevents these changes.

When an extract is judged to be sufficiently evaporated, the source of heat should be removed, and a small quantity taken out, placed on a cold plate in a cool place, and left for some time to thoroughly cool ; it may then be examined, and evaporation continued if not hard enough. Extracts of the Pharmacopœia are ordered either hard, which can be powdered, or of pilular consistence. One is ordered to be in powder, viz. Extractum Euonymi Siccum.

Special care should always be taken to avoid the "baking" of extract at the edges of the evaporating liquid, by assiduous stirring. Evaporation *in vacuo* is to be recommended, finishing in an open water or steam bath to get the right consistence.

The object of an extract being essentially to fully represent the activity of a drug in a very small compass, and in the natural condition, the following points should be attended to:

1. Thorough exhaustion of the drug.
2. Rapid manipulation throughout, to avoid decomposition.
3. Employment of lowest temperature compatible with rapid work.
4. Removal of inert and undesirable matter: *e. g.* if cold water be used, bring to boiling point to remove albumen; in some cases also precipitate mucilage with spirit.
5. Use of such menstrua that will extract the whole of the active ingredients with the least proportion of inert matter.

The student should prepare small quantities of certain of the extracts, so as to become perfectly familiar with the processes employed; the following are given as types of these preparations:

"*Extractum Gentianæ.*

"Take of—

Gentian root, sliced . . .	1 pound or 1 part.
Boiling distilled water . . .	1 gallon or 10 parts.

"Infuse the gentian in the water for two hours; boil for fifteen minutes; pour off, press, and strain. Then evaporate the liquor by a water-bath until the extract is of a suitable consistence for forming pills."—B. P.

The gentian is ordered to be "sliced" because, being of a soft and readily permeable nature, the water can extract the soluble constituents from small pieces practically as perfectly as from the powder; in this condition it presents the advantage of more ready removal of the liquor by straining. The first infusion for two hours thoroughly swells and softens the root, so that the subsequent boiling effectually exhausts it; boiling water being used, the albumen is not dissolved, and consequently no subsequent coagulation by heat is needed.

Extractum Papaveris.

"Take of—

Poppy capsules, freed from the seeds and in No. 20 powder	1 pound.
Rectified spirit	2 fl. ounces.
Boiling distilled water	a sufficiency.

"Mix the poppy capsules with two pints of the water, and infuse for twenty-four hours, stirring frequently; then pack in a percolator, and, adding more of the water, allow the liquor slowly to pass until about a gallon has been collected, or until the residue is exhausted. Evaporate the liquor by a water-bath until it is reduced to a pint, and when cold add the spirit. Let the mixture stand for twenty-four hours, then separate the clear liquor by filtration, and evaporate this by a water-bath until the extract has acquired a suitable consistence for forming pills."—B. P.

The seeds are separated because they contain much fatty oil, which would dilute the extract and render it of a greasy nature, as well as impede evaporation by floating on the surface of the water. The addition of the spirit causes precipitation of some gummy matter.

Extractum Aloës Barbadensis.

"Take of—

Barbadoes aloes, in small fragments .	1 pound or 1 part.
Boiling distilled water . . .	1 gallon or 10 parts.

"Add the aloes to the water, and stir well until they are thoroughly mixed. Set aside for twelve hours, then pour off the clear liquid, strain the remainder, and evaporate the mixed liquors in a current of warm air to dryness."—B. P.

Some resin and general impurities, as pieces of vegetable fibre, &c., remain undissolved; strain through flannel. The evaporation is best carried out *in vacuo*, but failing this the liquor should be exposed in very shallow vessels in the drying cupboard, which may be at about 38°—49° C., and should have a good draught of warm and dry air. A low temperature is employed, as a long-continued heat decomposes aloin in watery solution.

Ext. Aloës Socotrinæ is made by the same process.

“*Extractum Belladonnæ Alcoholicum.*

“Take of—

Belladonna root, in No. 20 powder	1 pound.
Rectified spirit	2 pints.
Distilled water	a sufficiency.

“Mix the belladonna with two pints of spirit, and macerate in a closed vessel for forty-eight hours; then transfer to a percolator, and when the fluid ceases to pass continue the percolation with water until two pints of liquid have been collected. Evaporate the percolated liquid by a water-bath until the extract has acquired a suitable consistence.” —B. P.

This macero-percolation is far better replaced by simple percolation, using at first just sufficient spirit to moisten the powder. Water is used to displace the spirit. (For a note on this see p. 225.) Extract. Gelsemii Alcoholicum is prepared in the same way.

Extractum Lupuli.

“Take of—

Hop	1 pound.
Rectified spirit	1½ pints.
Distilled water	1 gallon.

“Macerate the hop in the spirit for seven days, press out the tincture, filter, and distil off the spirit, leaving a soft extract. Boil the residual hop with the water for one hour, press out the liquor, strain, and evaporate by a water-bath to the consistence of a soft extract. Mix the two extracts, and evaporate at a temperature not exceeding 140° F. (60° C.) until it has acquired a suitable consistence for forming pills.”—B. P.

The treatment with spirit first is employed to secure the solution of the volatile oil and resin, but practically a considerable proportion of this is left behind in the pressed hop. The boiling with water removes the resin, forming a very turbid liquid, which should be strained as directed, not filtered. The final evaporation is conducted at 60° C., to avoid as much as possible the loss of volatile oil.

Extractum Belladonnæ.

Take of fresh leaves and young branches of belladonna, any quantity. Bruise in a stone mortar (or crush by rollers or mill) and press out the juice (as usual, see Chapter IX), heat the expressed juice gradually to 130° F. (54·4° C.), whereby the green colouring matter is precipitated (coagulated); separate this by a calico strainer. Heat the strained liquor to 200° F. (93·3° C.) to precipitate (coagulate) the albumen, keeping it at this temperature for five or ten minutes, and then filter through calico. Evaporate the filtrate by a water-bath to the consistence of a thin syrup; then add to it the green colouring matter previously separated and passed through a hair sieve; and, stirring the whole together assiduously, continue the evaporation at a temperature not exceeding 140° F. (60° C.) until the extract is of a suitable consistence for forming pills.

Note the object of each step in above process. Bruising or crushing—to break up plant tissues; pressure—to remove juice; heat to 54·4° C.—to coagulate chlorophyll; straining—to separate this before application of a greater heat, which would injure the colour; heat to 93·5° C.—to coagulate albumen; straining—to remove this, as its presence tends to render the finished extract liable to become mouldy; evaporation—to concentrate; rubbing chlorophyll through hair sieve—to finely divide the coagulum; evaporation at 60° C.—to concentrate without injury to colour.

Extractum Taraxaci.

"Take of fresh dandelion root, any quantity. Crush the root, press out the juice, and allow it to deposit; heat the liquor to 212° F. (100° C.), and maintain this temperature for ten minutes; then strain, and evaporate by a water-bath at a temperature not exceeding 160° F. (71·1° C.) until the extract has acquired a suitable consistence for forming pills." —B. P.

The juice is set aside to deposit earthy matter which has escaped removal by washing; also inuline, a substance allied to starch. The first heating to 100° C. coagulates albumen, which is removed by straining through calico. Extract of taraxacum is usually prepared late in autumn or early in winter, before the frosts set in; the yield is largest at this time, but the author considers that the best extract is produced in April or May, when the root possesses greater bitterness. The same remark applies to the *Succus.*

Table of Official Extracts.

Name of extract.	Menstruum.	Part of plant used.	Method of extraction.	Remarks.
Extractum Glycyrrhizæ	Cold water	Root	Double maceration	Note 1
,, Krameriæ	,,	,,	Macero-percolation	
,, Opii	,,	Inspissated juice of poppy	Triple maceration	Note 2
,, Quassiæ	,,	Wood	Macero-percolation	Note 3
,, Aloës Barbadensis	Boiling water	Inspissated juice of leaf	Infusion	P. 221
,, ,, Socotrinæ	,,	,,	,,	
,, Anthemidis	,,	Flower heads	Decoction	Note 4
,, Gentianæ	,,	Root	Infusion and decoction	P. 220
,, Hæmatoxyli	,,	Wood	,,	Note 5
,, Papaveris	,,	Fruit, without seeds	Infusion and percolation	P. 221
,, Pareiræ	,,	Root	,,	
,, Belladonnæ Alcoholicum	Rectified spirit	,,	Macero-percolation	Note 7
,, Cannabis Indicæ	,,	Flowering or fruiting tops	Maceration	Note 6
,, Gelsemii Alcoholicum	,,	Root	Macero-percolation	Note 7
,, Physostigmatis	,,	Seeds	,,	Note 8
,, Nucis Vomicæ	S. V. R. 4 parts, water 1 part	,,	,,	Chap. XXIII
,, Calumbæ	Proof spirit	Root	Double maceration	Note 9
,, Colocynthidis Comp.	,,	Fruit pulp	Maceration	Note 10
,, Jaborandi	,,	Leaves	Macero-percolation	Note 7
,, Stramonii	,,	Seeds	Percolation	Note 11
,, Euonymi Siccum	S. V. R. 1, water 1	Bark	,,	Note 12
,, Jalapæ	S. V. R., then cold water	Tuber	Maceration	Note 13
,, Lupuli	S. V. R., then boiling water	Strobiles	Maceration and decoction	P. 222
,, Cascaræ Sagradæ	Proof spirit, then cold water	Bark	Macero-percolation	Note 14
,, Rhamni Frangulæ	,,	,,	,,	,,
,, Rhei	,,	Root	,,	Note 15
,, Mezerei Æthereum	S. V. R., then solution in ether	Bark	Double maceration in S.V.R., then maceration in ether	Note 16

The following are prepared from fresh juices :

Name of extract.	Part of plant used.	Method of extraction.	Remarks.
Extractum Aconiti . .	Fresh leaves and flowering tops	Pressure	Note 17
„ Belladonnæ .	Fresh leaves and young branches	„	P. 222.
„ Conii . .	„	„	Note 17
„ Hyoscyami .	Fresh leaves, young branches, and flowering tops	„	„
„ Lactucæ . .	Flowering herb	„	„
„ Colchici . .	Fresh corm	„	Note 18
„ „ Aceticum	„	„	„
„ Taraxaci . .	Fresh root	„	P. 223

Note 1.—Prepared like the liquid extract, but evaporated to pilular consistence without addition of spirit. The addition of a small quantity of ammonia, as in U. S. P., facilitates extraction of glycyrrhizin.

Note 2.—Cold water used because boiling water would extract narcotine, to which the headache following the use of opium has been attributed. See Chapter XXIII.

Note 3.—Cold water used to avoid extraction of a gelatinous principle; the liquor is filtered when reduced to small bulk to remove a little albumen.

Note 4.—To every pound of drug used, 15 minims of oil of chamomile are incorporated with the extract while warm to replace that lost during the decoction and evaporation. The flower-heads are boiled with 10 parts of water until reduced to one half; a steam-bath should be used.

Note 5.—No iron vessels may be used for this preparation, or it will be discoloured by action of the peculiar tannic acid.

Note 6.—Spirit pressed out, recovered by distillation, and residue evaporated to soft extract.

Note 7.—Prepared exactly like alcoholic extract of belladonna; and, like it, if the water be allowed to pass into the alcoholic percolate, the character of the extract will be greatly altered by the presence of much mucilage.

Note 8.—Like Cannabis Ind., but by percolation. The evaporation, as well as distillation, is to be carried out in the retort to avoid unnecessary exposure to air whilst hot, which tends to oxidise the physostigmine, producing rubreserine. A better plan is to evaporate *in vacuo*.

Note 9.—Calumba is "cut small," not powdered, which would render straining difficult owing to starch. The B. P., 1867, ordered cold water for extraction, but so made the extract did not keep.

Note 10.—Colocynth pulp, 6 parts, is extracted by maceration for four days with proof spirit 160 fl. parts, the tincture pressed out and distilled; to the watery residue are added extract of Socot. aloes 12 parts, resin of scammony 4 parts, powdered curd soap 3 parts, and the whole evaporated by a water-bath till it has acquired a pilular consistence, adding powdered cardamoms when nearly completed. The seeds are removed from colocynth, as they are very oily; the cardamoms added just at end to avoid loss of aroma.

Note 11.—Ether is used to remove oil previous to percolation with proof spirit. This has been shown by Gerrard to be quite unnecessary, the extract made by direct extraction with proof spirit, and finally washing the extract with a little ether, being equally as strong.

Note 12.—The percolate is measured, and the amount of dry solid matter it contains is estimated by evaporating a known quantity, *e. g.* 5 or 10 cubic centimetres, in a weighed dish upon the water-bath, until the residue is quite hard; the dish and contents are weighed, and from these data the quantity of extract in the whole may be calculated.

For example :—Total volume of percolate = 1 gall. 2 pints = 200 fl. oz.
Weight of dish and dried residue from 5 c.c. = 23·308 grms.
„ „ alone . . . . . = 22·958 „
„ residue from 5 c.c. . . . = 0·350 „
20
„ „ 100 c.c. . . . = 7·000 „
= 7 per cent.

Therefore the whole volume (200 fl. oz.) will contain 7 oz. × 2 = 14 oz.

Having thus ascertained the extractive, one fourth this weight of sugar of milk is dissolved in the liquid after distilling off the spirit, and the whole evaporated on a water-bath until the extract becomes brittle on cooling. It is then powdered, and kept in well-closed bottles. So prepared, it is liable to "cake" together on keeping; it is better to add the powdered sugar of milk after the extract has been dried and powdered. The use of a weak spirit avoids the extraction of an oily substance which is taken out by strong spirit, and prevents pulverisation.

Note 13.—Like extract of hop, but macerated with cold water instead of by decoction.

Note 14.—Macerate with 2½ fl. parts of proof spirit, then percolate and displace with water, collecting 3¾ fl. parts, or till exhausted. Evaporate by water-bath.

Note 15.—Like Ext. Cascar. Sag., but using 3¾ fl. parts of proof spirit, and collecting 6¼ fl. parts, or till exhausted.

Note 16.—The bark is extracted by maceration in 7½ fl. parts of S. V. R. for three days, the liquid strained, again macerated in 2½ fl. parts for three days, strained and pressed. The filtered liquids are distilled, evaporated, and the extract so obtained macerated in a bottle in 1¼ fl. part of ether for twenty-four hours, the liquid decanted, distilled, and evaporated to a soft extract.

Note 17.—These are prepared like extract of belladonna.

Note 18.—Prepared like extract of taraxacum; for the acetic extract the crushed corms are mixed with acetic acid in the proportion of 6 fl. ounces to 7 pounds before expression. Colchicum contains much starch, which must be allowed to subside before evaporating the juice.

Questions on Chapter XIX.

1. Describe minutely the U. S. P. method of preparing fluid extracts by percolation. Which of the B. P. liquid extracts are made by this process?

2. Point out the special advantages of re-percolation, and the disadvantages of the ordinary methods when water is used to exhaust the drug.

3. Name the common constituents of drugs which are extracted by (*a*) ether, (*b*) cold water.

4. Describe briefly the preparation of the official liquid extracts of liquorice, coca, taraxacum, and fern.

5. Comment upon the official methods of preparing the liquid extracts of Rhamnus Frangula, ergot, cimicifuga, and sarsa.

6. Why is it necessary to manipulate rapidly in making extracts and similar preparations?

7. Describe minutely the preparation of extracts of conium and colchicum, giving reasons for each step of the processes.

8. What are the menstrua and methods for extraction of the following drugs?—Anthemis, Cannabis Indica, Calumba, Jaborandi, Krameria, Jalapa, Rhamnus Frangula, and Gelsemium?

9. Describe briefly the preparation of extracts of quassia, Socotrine aloes, rhubarb, euonymus, Calabar bean, compound colocynth.

10. Comment upon the official methods of preparing extracts of stramonium, belladonna (alcoholic), calumba, logwood.

11. Name the extracts prepared from fresh plants. Which are the "green" extracts?

12. What constituents are present in the finished extracts of belladonna, gelsemium, and gentian?

CHAPTER XX

EXTRACTION OF DRUGS (*continued*)

There remain several galenical preparations which are made by the methods adopted for the production of extracts, tinctures, &c. They include the official vinegars, three of the liniments, nine of the syrups, and some others.

Aceta—*Vinegars.*—Two of these are made with dilute acetic acid: that of *squill* (1 in 8) by maceration for seven days as for wines, but not making up the filtrate to full measure when finished; and that of *ipecacuanha* (1 in 20) by percolation with dilute acetic acid.

Vinegar of cantharides is made with a stronger acid: one part of the bruised drug is digested at 200° F. (93·3° C.) with a mixture of 1 fl. part of glacial acetic acid, and 6½ fl. parts of acetic acid for two hours, allowed to cool, and packed in a percolator, using acetic acid as menstruum until 10 fl. parts of percolate are obtained. The object of the addition of glacial acid is to increase the proportion of cantharidine in the product; the blistering effect of the flies depends upon cantharidine, which is not so readily soluble in 33 per cent. acid, as in 40 per cent. as ordered. The drug is ordered to be "bruised," not powdered; if used in powder percolation is almost impossible.

Vinum Ipecacuanhæ.—Acetic acid is also used to exhaust the drug for this preparation; 1 part of the root in about No. 20 powder should be moistened with about 1 part of dilute acetic acid for twenty-four hours (not in strong acid as officially ordered, because this renders the root so spongy and slimy as seriously to retard percolation), then packed in a percolator and percolation continued, first with 7 fl. parts more of diluted acid, and finally with distilled water till 20 fl. parts have passed through. These liquors (commencing with the weakest) are evaporated to dryness over a water-bath, the residue powdered, macerated in 20 fl. parts of sherry for forty-eight hours, agitating occasionally, and finally filtered.

Linimenta.—The three liniments are those of *soap*, *aconite*, and *belladonna*; the first of these is prepared by macerating 1 part of hard soap with ½ a part of camphor and $\frac{3}{16}$ fl. part of oil of rosemary in a mixture of distilled water 2, rectified spirit 8, for seven days at a temperature not exceeding 70° F. (21·1° C.), and filtration. The temperature is kept below 21° C. to avoid solution of stearate of sodium, which would be thrown out in a gelatinous condition on cooling.

Aconite and *belladonna* liniments are prepared by macerating 1 part of the root in No. 40 powder with 1 fl. part of rectified spirit for three days, stirring occasionally, then by percolation making up the volume to 1½ fl. parts after dissolving $\frac{1}{20}$ part of camphor in the percolate. Simple percolation is preferable.

Liquor Epispasticus is also prepared by percolation; 1 part of cantharides in powder is percolated with acetic ether until 4 fl. parts of percolate are obtained. Acetic ether is the best solvent of cantharidine; hence its employment for this preparation, which is intended as a very rapid vesicant.

Oleo-resina Cubebæ.—Cubebs in coarse powder is percolated with ether, the ether distilled off, the residue allowed to deposit fatty matter, and the liquid portion decanted. It is ordered in *coarse* powder because much of the volatile oil would be lost during the drying necessary before it could be reduced to *fine* powder.

Mistura Ferri Aromatica.—Four parts cinchona, 2 of calumba, 1 of cloves, and 2 of fine iron wire are macerated in 48 fl. parts of peppermint water for three days, then allowed to drain on a filter (or percolator), adding enough peppermint water to the contents of the filter to produce 50 fl. parts of filtrate. Finally add 2 fl. parts tincture of orange, and 12 fl. parts compound tincture of cardamoms. The finished preparation contains a small proportion of iron as organic salts (calumbate, kinate, &c.),

due to the natural acids of the drugs. It must be kept in a cool place (B. P.), or a deposit containing iron will be produced.

Syrupi.—Several of the syrups, which are prepared by simple processes, have been already considered; the remainder are made by dissolving sugar in a specially prepared liquid extraction of the drug.

In preparing this liquid the same care is needed to exclude albumen, &c., as is exercised in the case of extracts and fluid extracts, or the resulting syrups will certainly spoil; it is important also that the quantity of sugar used be so large that the sp. gr. of the resulting syrup shall be over 1·300, and in many cases over 1·330. This is necessary because weaker syrups undergo fermentation. The simple syrup of the B. P. consists of two parts of sugar dissolved in one part of distilled water, and its sp. gr. is 1·330. It would perhaps have been better had a sp. gr. 1·320 been aimed at, because the official syrup is so strong that sugar crystallises out in winter weather, and a syrup of sp. gr. 1·320 keeps well even in summer.

In the case of acid syrups, such as that of phosphate of iron, a sp. gr. of 1·300 to 1·310 is quite high enough; the syrup keeps free from fermentation, and if stronger than this there is some danger of the precipitation of part of the sugar as "grape-sugar," due to the gradual action of the strong phosphoric or other acid. On the other hand, such syrups as those of mulberry and poppy, which contain much vegetable matter, are peculiarly liable to fermentation, and should be made fully up to the gravity of 1·330; even then they will not keep long.

In some cases the B. P. directs that a small quantity of spirit shall be added to the syrups to assist in their preservation, but such an addition is of little value; very much more is necessary for the purpose. In two of these cases (mulberry and red poppy) it would be preferable to use about one half the amount of sugar with 15 to 20 per cent. of rectified spirit, so forming an *elixir*.

The following table includes the syrups not yet considered:

Name of syrup.	Part used.	Men-struum.	Method of exhaustion.	Strength when finished.	Sp. gr. (about).	Note.
Syrupus Hemidesmi	Dried root	Boiling water	Infusion for 4 hours	1 in 10 nearly	1·335	
* „ Limonis .	Fresh peel	Fresh juice	Infusion till cold	1 in 21 nearly	1·340	1
„ Mori . .	—	„	—	1 in 2 nearly, with S. V. R. 1 in 16	1·330	2
„ Papaveris .	Capsules without seeds	Boiling water	Infusion and percolation	1 in $2\frac{1}{4}$ nearly	1·330	3
„ Rhei . .	Dried root	S.V.R. 1, d. water 3	Percolation	1 in 15 nearly, with 1 of coriander	1·310	4
„ Rhœados .	Fresh petals	Hot water	Infusion for 12 hours	1 in $3\frac{1}{3}$ nearly, with $\frac{1}{5}$ of S. V. R.	1·330	5
„ Rosæ Gallicæ	Dried petals	Boiling water	Infusion for 2 hours	1 in 17 nearly	1·335	
* „ Sennæ .	Dried leaves	Water at 49° C.	Double digestion	1 in 2 nearly, with S.V.R. $\frac{3}{16}$	1·310	6
„ Tolutanus .	Balsam	Boiling water	Decoction for $\frac{1}{2}$ hour	1 in 29 nearly	1·330	7

Note 1.—The freshly expressed juice, one pint, is raised to the boiling point to coagulate albumen, and having added the peel, 2 oz., the whole is infused till cold, filtered through flannel, the sugar ($2\frac{1}{4}$ lbs.) dissolved by the aid of heat (see page 75). The final product should be allowed to stand two days, and be carefully filtered through paper pulp, when a better keeping preparation results.

Note 2.—Coagulate albumen, filter, dissolve the sugar, and add spirit.

Note 3.—$2\frac{1}{4}$ parts of poppies are exhausted as for extract (q. v.), and the liquor evaporated to $3\frac{3}{4}$ fl. parts; when cold 1 fl. part S. V. R. added to precipitate mucilage, and allowed to stand 12 hours, then filtered (through calico). The spirit is distilled off, the liquor evaporated to $2\frac{1}{2}$ fl. parts, and 4 parts sugar added.

Note 4.—Exhausted by slow percolation, liquid evaporated to 7 fl. parts, filtered, and sugar 12 parts added. Much of the aroma of coriander is lost during distillation and evaporation; the use of oil, as for syrup of senna, obviates this.

Note 5.—The water, 10 parts (not 20 parts, as ordered in B. P.), is heated on a water-bath; the petals, 12 parts, added and infused (not digested) twelve hours. The liquor is pressed out, the sugar, 36 parts, dissolved, and when nearly cold, $2\frac{1}{2}$ fl. parts S. V. R. added, finally making up the product to 58 parts by weight.

Note 6.—Digest 16 oz. of senna for twenty-four hours with 70 of water at 49° C.; press and strain. Digest marc with 30 of water, as before, for six hours; press and strain. Evaporate strained liquors to 10 fl. oz., and when cold add S. V. R. 3 fl. oz., with 3 minims of oil of coriander. Filter, and wash the filter with water until the filtrate measures 16 fl. oz. In this dissolve 24 oz. of sugar by the aid of heat. A higher temperature than 49° C. causes the solution of a quantity of mucilaginous matter, which renders the subsequent operations tedious without increasing the proportion of active principle (cathartic acid). Evaporation *in vacuo* is advantageous, as cathartic acid is injured by much heat, if adopted the liquor should first be brought to the boil to coagulate albumen. Spirit precipitates mucilage, which is removed by filtration; it would be better to wash with *weak spirit*, not *water*, as the latter must redissolve some of the mucilage. The coriander is used to flavour.

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* To be prepared by the student.

Note 7.—1¼ part of balsam is boiled for half an hour with 20 parts of water, enough water added to make the whole measure 16 fl. parts, and when cold, filtered. 32 parts of sugar are dissolved in the filtrate, and the whole made up to 48 parts with distilled water if necessary. The boiling water dissolves benzoic and cinnamic acids; the greater part of the latter crystallises out on cooling, and is removed by filtration.

Styrax præparatus is prepared by dissolving crude styrax in spirit, filtering from insoluble matter, and evaporating to remove spirit.

Ergotinum.

"Take of—

Liquid extract of ergot, Rectified spirit,	of each, equal fl. parts.

"Evaporate the fluid extract by a water-bath to a syrupy consistence, and when cold mix with the spirit. Let it stand for half an hour, then filter, and evaporate the filtered liquid to the consistence of a soft extract."—B. P.

Note.—This process yields varying results, according to the interpretation put upon the words "to a syrupy consistence." Literally it means that the *hot* liquid shall be of consistence of syrup; if it be so carried out, the yield will be very small. The better plan is to make ergotin direct from ergot, by exhausting it by means of a mixture of S. V. R. 2 fl. parts, water 1 fl. part, and evaporating to a soft extract, finally removing any *oil* by washing with ether.

Fel Bovinum Purificatum.

"Take of—

Fresh ox-bile	4 fl. parts.
Rectified spirit	q. s.

"Evaporate the bile to one fourth, and mix it with two fl. parts of the rectified spirit by agitation in a bottle, setting the mixture aside for twelve hours, or until the sediment subsides. Decant the clear solution, and filter the remainder, washing the filter and its contents with a little more of the spirit. Distil off most of the spirit from the mixed liquids, and evaporate the residue in a porcelain dish by the heat of a water-bath until it acquires a suitable consistence for forming pills."—B. P.

Note.—The addition of spirit causes the separation of mucus and epithelium, which render the preparation unstable.

There are many non-official preparations which are made by the processes of maceration and percolation; amongst the most important of these are the so-called "concentrated infusions." These are so made that when one fl. part is diluted with 7 fl. parts of distilled water the product shall represent the fresh infusion of the Pharmacopœia. Infusions made in this way, however, lack in most cases much of the aroma of the fresh preparation, and always contain a certain proportion of spirit, which is used as a preservative, and to render the aromatic principles soluble. The following formula is given as an example of these preparations.

Infusum Gentianæ Compositum Concentratum.

Take of—

Gentian root, in No. 16 powder	8 ounces.
Bitter orange peel ,,	8 ,,
Fresh lemon peel, cut small	16 ,,
Rectified spirit	24 fl. ounces.
Cold distilled water	24 ,,
Boiling distilled water	a sufficiency.

Mix the spirit with the lemon peel in a covered jar, macerate for three days, and press; to the marc add the cold water, macerate twelve hours and again press; mix the expressed liquors. With this liquid exhaust the gentian and orange by re-percolation, using the boiling water to displace the spirit until 64 fl. ounces of percolate have been obtained, and reserve this portion; now continue the percolation with boiling water until the percolate is only slightly coloured; evaporate to 16 fl. ounces, and mix this with the reserved portion. Allow to stand for three days, decant the clear liquor, and filter the remainder from the sediment.

For further information upon this subject the student may consult the pages of the 'Pharm. Journ.' (3) xviii, p. 615; xx. pp., 356, 486, 682.

Questions on Chapter XX.

1. Mention the strength, menstruum, and method of exhaustion employed for the following preparations:—Liquor Epispasticus; Mistura Ferri Aromatica; Acetum Scillæ; Acetum Cantharidis; Linimentum Aconiti.

2. Describe the preparation of ipecacuanha wine, with comments.

3. What is the object of the following?—Glacial acetic acid in Acet. Canthar.; rectified spirit in Syr. Mori; oil of coriander in Syr. Sennæ; temperature below 20° C. for Lin. Saponis; boiling of lemon and mulberry juices for syrups; use of S. V. R. for ergotin and ox-bile.

4. Describe the preparation of syrup of rhubarb.

5. Give the sp. gr. of syrups of rhubarb, tolu, hemidesmus, and rose.

6. Describe the official method of preparing ergotin, with criticisms.

7. Formulate your opinions respecting "concentrated infusions."

CHAPTER XXI

THE ISOLATION OF THE ACTIVE PRINCIPLES OF PLANTS

By the term "*active principle* of a drug" we mean that peculiar constituent upon which its medicinal efficacy depends. Some plants contain well-defined active principles, possessing striking or peculiar characteristics which render their isolation or detection comparatively simple; but our knowledge of the majority of drugs is at present too limited to admit of definite statements as to which is the active constituent, or indeed, in many cases, whether there be any *peculiar* active principle or not.

The active principles of drugs may usually be arranged under one of the following sections:

Alkaloids.—These are well-defined chemical substances, possessing more or less powerful basic properties, combining with the mineral and organic acids to form salts, many of which are crystalline. The free alkaloids usually react alkaline with litmus, methyl orange, and some other indicators; and their salts are commonly neutral, although a class of acid salts exists which contain double the amount of acid radical necessary for the production of a neutral salt. The alkaloids are *usually* insoluble or nearly so in water, but readily soluble in alcohol, chloroform, and less generally in ether; their salts, on the other hand, are commonly insoluble in ether and chloroform, but soluble in alcohol and water. Their aqueous solutions yield precipitates with chlorides of gold and platinum, potassio-mercuric iodide (Mayer's reagent), solution of iodine and iodide of potassium, bromine water, phospho-molybdic acid (Sonnenschein's reagent), phospho-tungstic acid, potassio-bismuthic iodide, tannic acid, picric acid, and less frequently with mercuric chloride.

Ammonia or the stronger alkalies liberate alkaloids from solutions of their salts. All alkaloids contain nitrogen, and usually oxygen also. Examples of plants the activity of which is due to an alkaloid or alkaloids are cinchona, nux vomica, aconite, belladonna, &c.

Glucosides.—These are compounds which possess less marked characteristics than the alkaloids; they are usually neutral, although frequently acid, and sometimes slightly basic; they are readily decomposed, with production of glucose or an allied sugar, and another substance which varies with the character of the glucoside, but is usually an alcohol, an aldehyd, or a hydrocarbon. This decomposition may be brought about by boiling with dilute sulphuric, hydrochloric, or other acid, or with alkalies; by treatment with suitable ferments; or in some cases by simple boiling with water, or heating with water to a high temperature in sealed tubes. The acid glucosides form more or less stable compounds with metallic bases, such as oxides of lead and barium. The glucosides commonly contain no nitrogen, although this element is sometimes present, and in some few cases sulphur also. They are usually soluble in water and alcohol, many are soluble in chloroform, but most are insoluble in ether. They are frequently crystalline.

Most glucosides reduce alkaline copper solution when boiled with it, and all do so after decomposition by dilute acids; all are precipitated by a solution of ammonic molybdate acidulated with hydrochloric acid, and some give precipitates with picric and tannic acids and other alkaloidal reagents.

Examples of drugs owing their activity to glucosides:—Digitalis, mustard, bitter almonds, senega, and strophanthus.

Tannins.—The tannins are nearly allied to the glucosides, being in most if not all cases either glucosides or the alcohols, or other products of the decomposition of glucosides. They are all more or less soluble in water, soluble in alcohol, but usually only slightly so in ether or chloroform. Their solutions, especially aqueous, are very liable to decomposition. Most of them give precipitates with alkaloids, gelatine, copper acetate, and tartarated antimony, and all do so with lead acetate.

Examples of official drugs:—Galls, krameria, kino, matico.

Resins.—Resins are characterised by marked insolubility in water (some are slightly soluble in boiling water), and ready solubility in spirit; towards ether, chloroform, and petroleum ether their behaviour is variable. They are usually brittle solids of a yellow or brown colour, in some cases are glucosides, and very frequently present marked acid characters, combining perfectly with the alkalies, &c., forming compounds allied to the soaps, from aqueous solutions of which a mineral acid commonly reprecipitates the original resin. The lead salts are almost always insoluble in water. Such resins are called *acid resins,* to distinguish them from neutral resins, which do not form alkaline salts by treatment with either aqueous or alcoholic alkalies. The resins melt at a gentle heat, and decompose when strongly heated.

Examples of resinous drugs:—Jalap, scammony, cascarilla, podophyllum, myrrh.

Gums.—Although these can scarcely be considered as active principles, it is nevertheless very common to find drugs whose medical usefulness depends largely upon the presence of *gum* or *mucilage.* A gum is exactly the opposite to a resin, being insoluble in alcohol, ether, chloroform, or benzin, but either wholly or partially soluble in water. In the latter case the whole swells up into a tenacious, almost jelly-like condition, which can be fairly evenly distributed throughout the liquid, and does not subside on standing. Drugs consisting of or containing much gum or mucilage are linseed, acacia, tragacanth, myrrh.

None of the gums are isolated by artificial means for use in medicine.

Many drugs contain both a resin or resins and mucilage or gum, the latter serving to emulsify the former when mixed with water. In some cases the dried-up juice of the plant consists mainly of resin and gum, and is the official form of the drug. Such combinations are called *gum-resins.*

Examples of gum-resins are asafœtida, myrrh, ammoniacum, scammony.

Essential oils.—These have been already described, with the methods of preparation (see p. 127).

Examples:—Anise, cloves, lavender.

Acids.—There are some plants which owe their activity to the febrifuge character of acids or acid salts, such as tartaric, citric, and malic acids ; these are in many cases fruits. Examples :—Lemon (juice), tamarinds.

Neutral principles.—This is a convenient name which is used to express any definite principle which cannot be classified under any of the above sections. Example :—Elaterin.

The following is a list of definite principles which are official in the B. P., either in the free state or as salts. In most cases they are the *active* principles of the plants.

	Source.		Source.
Alkaloids and alkaloidal salts :			
Aconitina . .	Aconitum napellus	Codeina . . .	Opium
		Morphinæ Acetas .	Opium
Atropina . . .	Atropa belladonna	,, Hydrochloras	
,, sulphas .		,, Sulphas .	
Beberinæ sulphas .	Nectandra Rodiæi	Physostigmina . .	Physostigma venenosum
Caffeina . . .	Camelia thea or Coffea arabica		
,, citras .		Pilocarpinæ Nitras .	Pilocarpus pennatifolius
Cinchonidinæ sulphas	Cinchona succirubra, &c.	Quininæ Hydrochloras	Cinchona succirubra, &c.
Cinchoninæ sulphas		,, Sulphas .	
Cocaina	Erythroxylon Coca	Strychnina . .	Strychnos nux vomica
,, hydrochloras			
Glucosides :			
Acidum Tannicum .	Galla	Salicin . . .	Salix alba, &c.
Resins :			
Jalapæ Resina .	Ipomœa purga	Resina . . .	Terebinthina
Podophylli Resina .	Podophyllum peltatum	Scammonii Resina .	Convolvulus scammonia
Acids :			
Acidum Benzoicum .	Benzoin	Acidum Oleicum .	Oils and fats.
,, Citricum .	Juices of Citrus limonis and C. bergamia	,, Salicylicum .	Betula lenta ; Gaultheria procumbens
,, Meconicum .	Opium	,, Tartaricum .	Argol
Neutral principles :			
Aloin . . .	Aloes	Santoninum . .	Artemisia maritima
Elaterinum . .	Ecballium elaterium	Picrotoxinum . .	Animirta paniculata
Chrysarobinum .	Andira araroba		
Stearoptenes :			
Camphor . .	Cinnamomum camphora	Thymol . . .	Thymus vulgaris, Carum ajowan, and Monarda punctata
Menthol . . .	Mentha arvensis and M. piperita		

In addition to these, two other alkaloidal salts are prepared from other alkaloids, viz.: Apomorphinæ Hydrochloras from morphine, and Homatropinæ Hydrobromas from tropine.

The methods by which alkaloids and other active principles are commercially manufactured are usually kept secret by the makers; small quantities may, however, be prepared by the methods of the B. P., or modifications of them, some of which will now be explained.

Alkaloids.—The methods by which alkaloids are prepared vary with the drug operated upon, the actual method employed being determined by the physical and chemical characteristics of the alkaloid and of the accompanying plant constituents, which for the purposes of this operation must be regarded as *impurities.* The methods, however, are divisible into three stages:

1st. The extraction of the drug by means of a suitable solvent.

2nd. The separation of crude alkaloid.

3rd. The purification of this crude alkaloid from colouring matter and other impurities.

1. The solvent employed should be that liquid which most effectually removes the *alkaloid* from the drug with as little as possible of the accompanying plant constituents, such as oils, resins, mucilage, &c. Sometimes the powdered drug is treated directly with the solvent, thus extracting the alkaloid in its natural condition; at others the drug is first treated with milk of lime or magnesia and dried, then exhausted with the menstruum. The action of the lime is to liberate the alkaloid from its natural salts, and also frequently to render the accompanying acids, colouring matters, and resins, &c., insoluble in the menstruum employed.

2. The separation of the crude alkaloid is effected in many ways; it is often first necessary to remove the menstruum by distillation (always if spirit be used for extraction), with or without the addition of an acid; the extract is then mixed with water and filtered, whereby much resinous or oily matter is removed; it is advantageous to employ pumice or sand to aid the filtration, these substances preventing the oily matter from choking the pores of the paper. The filtered liquid is then treated either by precipitation or by the method of

separation to remove the crude alkaloid or alkaloids. For the precipitation ammonia, sodium carbonate, or other alkali is commonly employed, which separates the alkaloid in a free state, or in some cases tannic acid, picric acid, or iodine may be used; in any case the precipitate so produced is removed by a filter and washed. Where ammonia or other alkali is used the washed precipitate constitutes *crude alkaloid,* but in the case of the other compounds the precipitates require treatment by special methods. The compounds with tannic or picric acid may be decomposed by treatment with barium hydrate, lead oxide, magnesia, or ammonia, whereby the alkaloid is liberated, which is dissolved in alcohol, ether, chloroform, or other suitable solvent; this solution on evaporation or crystallisation yields the *crude alkaloid.* The compounds with iodine are decomposed by thiosulphate of sodium or sulphurous acid, and the liberated alkaloid washed with water, or dissolved and separated by the method of "agitation" or "separation." The process of *separation* or *agitation* consists in agitating the acid solution of the alkaloids with chloroform, ether, benzin, or other solvent immiscible with water; this dissolves out chlorophyll, fatty and resinous substances; on allowing the mixture to stand it separates into two layers, which are separated by a tapped funnel or *separator,* two forms of which are shown in Figs. 66 and 67. When chloroform is employed it forms the lower layer; when ether, the upper. This agitation and separation should be continued with successive portions of solvent until no more colour is imparted to it. The solvent so used should be washed once with water, and the washings added to the acid liquid, which is then rendered alkaline by ammonia, soda, or sodium bicarbonate, and again agitated with more solvent. The alkali liberates the alkaloid, which dissolves in the chloroform or other solvent, and after standing to form two layers this may be removed, and the crude alkaloid obtained by distilling off the solvent.

FIG. 66.

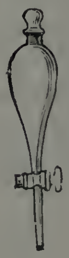
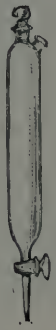


FIG. 67.



3. *The purification* of the crude alkaloid obtained by

either method is the most difficult part of the operation, and is accomplished by various methods, among which may be mentioned (1) successive crystallisations from alcohol, ether, chloroform, &c.; (2) conversion into salts, such as hydrochlorate, tartrate, nitrate, &c., and crystallisation from alcohol or water; (3) decolorisation by animal charcoal, for which purpose a spirituous solution of the alkaloid or an aqueous or spirituous solution of one of its salts is gently warmed with the finely powdered charcoal, allowed to stand some time, and filtered, after which the alkaloid is recovered from the liquor by precipitation or crystallisation; and (4) methods of precipitation, either from aqueous solution of the salts by means of a different precipitant from that used in the first separation, *or* from a solution of the alkaloid in alcohol, ether, or chloroform by the addition of another liquid in which the alkaloid is insoluble—*e. g.* an impurity accompanying emetine is precipitated from ethereal solution by petroleum benzin; *or* emetine itself is separated from its ethereal solution by passing *dry* HCl, which precipitates the base as hydrochlorate. It is often necessary to combine two or more of these processes. Very frequently, also, the crude alkaloid consists of two or more alkaloids, in which case a separation must be effected; for this purpose similar methods to those above mentioned are employed.

We shall now describe the methods of preparation of the more important B. P. alkaloids. The student should prepare two or three of these; perhaps the best for his purpose are quinine, strychnine, and atropine.

Aconitina, B. P.—Aconite root is exhausted with spirit 56 o. p. by maceration and percolation, the spirit recovered by distillation, and the last portions removed by evaporation. The residual extract, which consists of the alkaloidal salts, resins, colouring matter, sugar, and oil, is mixed with twice its weight of boiling water, cooled, and filtered through paper; the filter retains much oil and resinous matter, the liquid containing alkaloidal salts, sugar, and colouring matter. To the filtrate add slight excess of solution of ammonia, and heat gently over a water-bath. Separate the precipitate on a filter and dry it. This precipitate consists of crude alkaloids containing some colouring matter mechanically carried down with the precipitate. This precipitate is dried, reduced to a fine powder and macerated with several successive small portions of *pure* ether. The ether dissolves *aconitine*, leaving aconine and some other impurities insoluble. The ethereal solution is distilled and the extract dried, dissolved in warm distilled water slightly acidulated with sulphuric acid; when cold, dilute solution of ammonia is slowly added until in *faint* excess. The precipitated alkaloid is collected on a filter, washed with the least possible quantity of distilled water, and dried by pressure between folds of bibulous paper and subsequent exposure to the air. N.B.—A considerable loss of alkaloid occurs in the washings. Mr. Williams has described a method of preparation whereby a purer, crystalline alkaloid is obtained (*vide* 'P. J.' [3], xviii, p. 238; also Rogers and Richards, 'C. and D.,' i, 1891, p. 205).

Apomorphinæ Hydrochloras.—This is obtained from morphine or codeine by heating in sealed tubes with hydrochloric acid:

$$C_{17}H_{19}NO_3 - H_2O = C_{17}H_{17}NO_2.$$
$$C_{17}H_{17}NO_2 + HCl = C_{17}H_{17}NO_2HCl.$$

Atropina.—Belladonna root is exhausted by maceration and percolation with spirit 56 o. p. The resulting percolate is treated by agitation with slaked lime, whereby the alkaloidal salt (malate?) is converted into calcium salt, with liberation of free alkaloid, which remains in solution. Some colouring matter as well as malate of calcium is precipitated. The liquid is filtered, diluted sulphuric acid added in *very* slight excess, whereby the alkaloid is converted into sulphate, and a great portion of the excess of lime precipitated as sulphate; the liquid is again filtered and three fourths of the spirit distilled off, the residue mixed with about one sixth its volume of water, and the distillation and evaporation continued *rapidly* until all alcohol is dissipated. The alkaloid is converted into sulphate before distillation because that salt is less readily acted upon by heat than the free base; the water is added to avoid heating the extract with strong sulphuric acid, which would occur when all the spirit had been removed. When cool, *exactly* neutralise with a solution of potassium carbonate. Set at rest for six hours, then filter, thus removing fatty and resinous bodies; then add potassium carbonate to a decided alkaline reaction. The liquid is agitated with chloroform, which dissolves the precipitated alkaloid, and when settled the chloroform is separated from the supernatant aqueous liquid. The chloroform is recovered by distillation, leaving crude alkaloid, which is dissolved in a small quantity of rectified spirit, the solution digested with a little animal charcoal, filtered, evaporated to a low bulk, and cooled till colourless crystals are obtained. Or the crude alkaloid may be dissolved in an excess of dilute HCl, a solution of iodine and iodide of potassium added, the precipitate collected on a filter, washed, decomposed by thiosulphate of sodium, and the alkaloid extracted from the filter and solution by agitation with chloroform, and the chloroform solution mixed with ether or benzin and allowed slowly to evaporate, when the residue will be crystalline.

Atropinæ Sulphas.—Vide p. 178.

Beberinæ Sulphas.—Bebeeru bark (*Nectandra Rodiæi*) is exhausted by maceration and percolation with distilled water acidulated with sulphuric acid, the percolate concentrated, milk of lime added till the liquid is nearly neutralised but still retains a *distinctly* acid reaction, and after two hours filtered through calico, whereby much sulphate of calcium and colouring matter are removed, the precipitate washed with cold water, and the crude alkaloids precipitated by the addition of dilute ammonia in slight excess. The alkaloids are collected on a cloth, washed with a little water, squeezed gently, and dried on a water-bath. The dried precipitate is powdered, boiled with successive small quantities of rectified spirit until no more soluble matter is removed. The spirit contains the alkaloids beberine and nectandrine, leaving some colouring matter insoluble. The united spirituous liquors are mixed with a little water and distilled to recover the spirit. To the residue is added slowly diluted sulphuric acid to a faintly acid reaction; then the whole is evaporated to dryness and pulverised; cold distilled water slowly added, stirring diligently, the liquid filtered, the filtrate evaporated to a syrup, spread upon glass plates, and scaled at a temperature not exceeding

60° C. The preparation consists of a mixture of sulphates of two or perhaps several alkaloids.

Caffeina (Theine).—The B. P. gives no definite directions for its preparation; it may be obtained, however, by exhausting tea or coffee by means of boiling water, separating tannin and colouring matter by agitation with lime, filtering, and then adding subacetate of lead and again filtering, keeping the solution hot. Excess of lead is separated by means of sulphuretted hydrogen or sulphuric acid and filtration, the filtrate concentrated to a low bulk and allowed to crystallise; the crude caffeine so obtained is purified by solution in spirit, chloroform, or water, and recrystallisation.

Or it may be made by treating powdered tea with milk of lime, drying at a low temperatue, and exhausting by percolation with boiling spirit. On distilling off the spirit the crude caffeine is left, together with a little oily and colouring matter; it may be purified by dissolving in boiling water, from which it crystallises out on cooling.

Caffeinæ Citras.—See p. 118.

Cinchonidinæ Sulphas
Cinchoninæ Sulphas } .—See Quininæ Sulphas, p. 247.

Cocainæ Hydrochloras.—The coca leaves in fine powder (No. 40 to 60) are exhausted by repercolation with methylated spirit containing about $\frac{1}{500}$ part of sulphuric acid or double that quantity of hydrochloric acid. The percolate contains sulphate or hydrochlorate of the alkaloids, together with chlorophyll, tannic and other vegetable acids, sugar, &c.; it is distilled, and to the hot liquid is added 2 per cent. sulphuric or hydrochloric acid in quantity equal to about $\frac{1}{10}$ of the original weight of leaves. Ether is added, agitated, and after separation the aqueous liquid containing the alkaloids is removed, the ethereal solution of chlorophyll being washed twice with dilute acid. The watery solution having been washed with ether until the latter comes off nearly free from colour, to the mixed aqueous liquids ether is added and a considerable excess of carbonate of sodium, whereby the alkaloids are liberated from the acid and dissolved by the ether. The separated aqueous liquid is in turn washed with ether, and the mixed ethereal liquids agitated with several small quantities of dilute hydrochloric acid. The solution of hydrochlorates (which must contain no *free* acid) so obtained is decolourised by percolation through pure animal charcoal, the alkaloids separated by sodium carbonate and ether as before, the crude cocaine dissolved and crystallised several times from alcohol, exactly neutralised with HCl, evaporated to dryness, and finally dissolved in the smallest possible quantity of *absolute* alcohol, the solution filtered if necessary, and six times its volume of *anhydrous* ether added, which causes the precipitation of hydrochlorate of cocaine nearly free from the other alkaloids which accompany it. A repetition of this process of solution in alcohol and precipitation by ether will give the salt in a state of purity.

Codeina.—Vide Morphinæ Hydrochloras.

Homatropinæ Hydrobromas.—The hydrobromate of an alkaloid prepared from tropine.

Morphinæ Hydrochloras.—One pound opium is exhausted by three successive macerations with 2 pints of cold water, and strong expression. The mixed liquors evaporated to *one* pint and strained. Chloride of calcium is now added, which by double decomposition produces meconate and sulphate of calcium, and hydrochlorates of some of the alkaloids; the mixture is evaporated until upon cooling it becomes solid from crystallisation of morphine hydrochlorate, &c. The mass is pressed strongly, preserving the dark-coloured liquid which exudes. The pressed cake is triturated with half a pint of boiling water, which dissolves the morphine hydrochlorate, leaving most of the calcium sulphate and meconate, and some colouring matter. The whole is thrown on a paper filter and the residue washed with boiling water. The filtered fluids are evaporated, cooled, allowed to solidify, and pressed again, and if the pressed liquid be still highly coloured the whole operation is repeated a third time, the expressed liquids being always preserved. The pressed cake is finally dissolved in six fl. ounces of boiling distilled water, animal charcoal added, digested for twenty minutes, filtered, the charcoal and filter washed with boiling water, and ammonia in small excess added to the filtrate. The crystalline precipitate which forms is morphine, and is washed with cold water until free from ammonic chloride. The dark expressed liquids may be further concentrated and made to yield an additional crop of crystals, which by pressure, re-solution, &c., may be purified as before.

The precipitated morphine so obtained is diffused through 2 fl. ounces of boiling distilled water and diluted hydrochloric acid cautiously added, with constant stirring, until the morphine is all dissolved and a neutral solution obtained. Upon cooling, crystals of morphine hydrochlorate separate, which may be collected, drained, and dried on filtering paper. The mother liquor yields a further crop of crystals by evaporation and cooling.

Morphinæ Acetas.—This is prepared from the hydrochlorate by precipitation with ammonia, collecting and washing the morphine, diffusing in boiling water, adding acetic acid to dissolve it. The solution is evaporated upon a water-bath, maintaining the acetic acid in slight excess (by occasional addition of a few drops) until it concretes on cooling. Lastly, dry the salt with slight heat, so as to avoid much loss of acetic acid, and reduce to powder.

Morphinæ Sulphas.—This salt is prepared by diffusing the morphine obtained as described under "Morphinæ Hydrochloras in about twice its weight of boiling water, and adding to the fluid, kept hot, diluted sulphuric acid, gradually and with constant stirring, so that the morphine may be entirely dissolved, and a neutral solution obtained. Set aside to cool and crystallise. Drain the crystals, and dry them on filtering paper. By further evaporating the mother liquor, and again cooling, additional crystals are obtained."—B. P.

Codeine is obtained from the ammoniacal liquors from which the morphine has been separated, by agitation with chloroform, evaporating the chloroformic solution to dryness, treating the residue with water, in which codeine dissolves, precipitating with caustic potash, and purifying the precipitated alkaloid by recrystallisation from ether.

Physostigmina.—This alkaloid is prepared by a method similar to that used for cocaine, without the final solution of hydrochlorate in alcohol and precipitation by ether.

Pilocarpinæ Nitras.—Jaborandi is exhausted by means of a mixture of spirit 56 o. p. 2 parts, water 1 part, containing about ½ per cent. of hydrochloric acid; the solution distilled and evaporated to small bulk, ether added, and the watery liquid separated. This solution, after washing with ether, is treated with chloroform and ammonia or carbonate of sodium, the chloroformic solution distilled, the residual crude alkaloid converted into nitrate, and purified by means of animal charcoal and recrystallisation.

Quininæ Hydrochloras.—Prepared from the same sources and by the same processes as sulphate of quinine, the separated alkaloid being neutralised by hydrochloric acid.

Quininæ Sulphas.—The most important alkaloids present in the barks of the various species of Cinchona and Remijia used for their preparation are quinine, cinchonidine, cinchonine, quinidine, cupreine (in Remijia), and the so-called amorphous alkaloid, which probably consists of several. Sulphate of quinine for medicinal use is officially required to contain not "much more than 5 per cent. of the sulphates of other alkaloids." The following is a method of preparation:

The bark is reduced to powder, mixed with milk of lime containing CaO equal to about one sixth the weight of the bark employed. These are allowed to remain in contact for about twenty-four hours, so that the whole may be uniformly damp. It is then packed in steam-jacketed percolators, and exhausted by means of hot fusel oil from which the fractions of lower boiling point have been chiefly removed by steam distillation. The percolate contains the alkaloids with some colouring matter, and is introduced into a large separator, thoroughly agitated while warm with successive small quantities of hot dilute hyrochloric acid, until the whole is extracted from the fusel oil, which may be used for a succeeding operation after being distilled. The yellow aqueous solution of alkaloidal hydrochlorates is now treated with ether, and a considerable excess of solution of sodium hydrate. The ethereal liquid will then contain the quinine and amorphous alkaloid and some quinidine; the alkaline liquid retains cupreine; and the precipitate consists of cinchonine, cinchonidine, and some quinidine. The alkaline solution and the precipitate are washed two or three times with ether, and the washings added to the original ethereal liquid, which in turn is washed once with water. The ethereal solution is now distilled, and the residue treated with several small quantities of hot dilute sulphuric acid until dissolved; the solution exactly neutralised by the addition of soda, and set aside to crystallise; the crystals of crude sulphate of quinine so obtained are redissolved in boiling water, passed through a column of pure animal charcoal, and further purified by crystallisation. The mother liquors will yield a further crop of crystals by evaporation. The precipitate which was separated from the ether is dissolved in hot dilute H_2SO_4, neutralised by NaHO, and set aside to crystallise. The crystals are crude sulphate of cinchonidine, and may be purified by recrystallisation; the mother liquors are evaporated for a further crop of crystals, and each lot is purified by successive crystallisations, whereby the alkaloidal sulphates are separated. Or the original solution of sulphates of these alkaloids not dissolved by ether is freed from quinidine by cautious addition of potassium iodide, with the addition of a small proportion of spirit, the liquid filtered, and the filtrate used for obtaining sulphates of cinchonidine and cinchonine by successive crystallisations; or cinchonidine may be precipitated as tartrate.

Strychnina.—Nux vomica seeds, 1 part, are split, dried at 100° C., and reduced to fine powder. The powder is digested for twenty-four hours at a gentle heat with 2½ fl. parts of spirit 56 o. p., mixed with half its volume of water, strained, pressed, and the digestion, &c., repeated twice. The spirit is distilled, the aqueous residue evaporated to 1 fl. part, and filtered when cold; some oily and resinous matter is thus removed. To the filtrate solution of lead acetate is added so long as it occasions a precipitate, then filtered; the precipitate washed with cold water, adding the washings to the filtrate. The lead acetate precipitates much colouring matter and igasuric acid, the alkaloids strychnine and brucine being converted into acetates. The solution is evaporated to ½ fl. part; when cold, ammonia is added in slight excess, stirring thoroughly. The mixture is allowed to stand twelve hours, when the precipitate of crude alkaloids (strychnine and brucine) is collected upon a filter, washed with a small quantity of cold distilled water, dried in the water-bath or air-oven, and boiled with successive portions of rectified spirit until the washings scarcely taste bitter. Distil off most of the spirit, evaporate to about $\frac{1}{32}$ part, and set aside to cool; the yellowish mother liquor is cautiously poured off (containing the bulk of the brucine) from the white crust of strychnine which adheres to the vessel.

The strychnine is washed upon a paper filter, with a mixture of 2 parts rectified spirit with 1 of water, till the washings cease to become red on the addition of nitric acid (a red colour indicates presence of brucine); finally, dissolved by boiling with $\frac{1}{8}$ part S. V. R., and set aside to crystallise. More crystals may be obtained by evaporating the mother liquor. N.B.—Strychnine is freely soluble in hot S. V. R., but very slightly so when cold. Brucine is readily soluble both hot and cold.

Veratrina.—Cevadilla is macerated with half its weight of boiling distilled water for twenty-four hours, pressed, and thoroughly dried in a warm place. It is now beaten in a mortar, and the seeds separated from the capsules by brisk agitation in a deep narrow vessel, or by winnowing. The seeds are ground in a mill, made into thick paste with spirit 56 o.p., and exhausted by percolation with spirit. The percolate, which contains veratrine, resin, oil, and colouring matter, is concentrated by distillation, until a deposit begins to form, when the residue is poured, whilst hot, into twelve times its volume of cold distilled water. This effects the separation of much resin and oil as a precipitate, which is removed by a calico strainer, and washed with water until the filtrate ceases to precipitate with ammonia. To the united filtrates, ammonia is added in slight excess, the precipitated alkaloid allowed to settle, the liquid removed by decantation and filtration, and the residue washed till the filtrate passes colourless. The precipitate is diffused in distilled water, and dissolved by diluted hydrochloric acid to a feebly acid reaction. Animal charcoal is added, the whole digested warm for twenty minutes, filtered, and allowed to cool. Ammonia is now added, the precipitate separated by subsidence and filtration, washed with water till free from chlorides, then dried by means of bibulous paper and a gentle warmth.

GLUCOSIDES.—The methods by which glucosides are prepared differ very greatly. An aqueous or weak spirituous extraction of the drug is usually taken as the starting-point, from which the glucoside may frequently be precipitated by means of subacetate of lead, the lead precipitate being washed and decomposed by sulphuretted hydrogen; or the precipitate is mixed with milk of lime and dried over a water-bath, the dried residue powdered and percolated with spirit, which on evaporation yields the glucoside.

Another method is to leave the drug in contact with milk of lime for twenty-four hours, dry, and extract the glucoside by percolation with a suitable solvent.

Still another method often yields good results. The drug is exhausted by percolation with spirit, or a mixture of spirit and water containing not less than 50 per cent. of spirit 56 o. p. This fluid extract is then treated with acetate of lead, and the precipitate removed by filtration. The filtrate is freed from lead by sulphuretted hydrogen, or preferably phosphate of sodium, and again filtered. This second filtrate is concentrated by distillation to remove spirit, and the glucoside removed by agitation with ether, chloroform, or other suitable solvent. The ethereal (&c.) solution is removed and distilled to obtain a dry residue, which may be further purified, if necessary, by crystallisation from spirit, ether, or other solvent.

There are but few glucosides official in the British Pharmacopœia, viz. tannic acid, salicin, and the resins of jalap and scammony. These latter two are considered under "Resins."

Acidum Tannicum.—Powdered galls is exposed to a damp atmosphere for two or three days (to absorb moisture), and sufficient ether then added to form a soft paste. (Tannic acid is insoluble, or nearly so, in pure ether, but readily soluble in ether saturated with water.) This paste is allowed to stand in a well-closed vessel for twenty-four hours, then enveloped in a linen cloth and submitted to powerful pressure, reserving the expressed liquid. The powdered cake is mixed with sufficient ether, to which one sixteenth of its volume of distilled water has been added, to form a soft paste, and again pressed. The expressed liquids are mixed and exposed to spontaneous evaporation until by the subsequent application of a little heat it has acquired the consistence of a soft extract; it is then placed on earthen plates or dishes, and dried in a hot-air chamber at a temperature not exceeding 100° C.

Salicinum.—The barks of several species of Salix and Populus contain this glucoside. For its preparation a decoction is prepared with hot water, tannin and colouring matter removed by lead acetate, and the excess of lead from the filtrate by cautious addition of sodium phosphate, avoiding large excess. The filtrate is evaporated, allowed to crystallise, the crystals purified by solution in hot water, and digestion with animal charcoal, and then recrystallised. Or it may be prepared by Erdmann's process as follows:—One pound of the bark is macerated for twenty-four hours with a gallon of water and two ounces of lime, then boiled for half an hour. The residue is again treated in the same way. The mixed decoctions having been cleared by decantation and filtration, the clear liquor is evaporated to 2 pints, digested with 8 ounces of animal charcoal, filtered, and evaporated to dryness. The residue is exhausted with spirit of sp. gr. ·845, and the tincture evaporated to crystallisation. The impure salicin so obtained is purified by re-solution and recrystallisation after treatment with animal charcoal. Besides mucilaginous and albuminous matters, tannin, &c., willow bark contains lactic acid, which remains in solution as calcium lactate when the salicin crystallises from the alcoholic solution.

Resins.—Resins are prepared by two principal methods:

1st. From natural oleo-resins, such as turpentine, by distilling off the volatile oil, when the resin remains in the still. This method is used in the preparation of Resina, B. P.

2nd. The drug is exhausted by percolation with spirit, a small quantity of water added, and the tincture distilled till all the spirit is removed; the residue is removed while still hot to a suitable vessel, and allowed to cool, after which the supernatant liquid is poured off (containing commonly sugars and colouring matters or tannins) from the resin, which is then washed with successive small quantities of warm water, and finally dried by a water-bath or stove. This process is used for Resina Jalapæ and R. Scammonii, B. P.

A slight modification of this method is employed in the case of Resina Podophylli, B. P. (Podophyllin). No water is added to the tincture, which is distilled at once, and when nearly all the spirit is removed the concentrated liquid is poured into a large volume of cold water. By this means the resin separates in a finely divided condition; it is allowed to stand for twenty-four hours, the supernatant fluid removed by decantation, the resin washed on a filter with distilled water, and finally dried.

Still another modification is sometimes employed; in this case the strong solution of resin, &c., obtained as in the case of resin of podophyllum, is poured into very dilute hydrochloric acid instead of water, the succeeding operations being the same. This method was official for the preparation of podophyllum resin in the 1867 Pharmacopœia (additions, 1874). The HCl causes a more perfect and ready separation of the resin.

Essential Oils.—These have been already described (*vide* p. 127).

Acids.—The properties of *acids* have been already described (p. 10); those which are included in this section are all of an organic nature; their methods of preparation are very various.

Acidum Benzoicum.—By sublimation (*vide* p. 133).

Acidum Citricum (*vide* p. 175).

Acidum Meconicum.—This acid is a bye-product in the preparation of morphine. The precipitate obtained with chloride of calcium is washed and treated with dilute sulphuric acid, whereby the calcium meconate is decomposed, with formation of calcium sulphate and meconic acid. The latter is dissolved by boiling water, and separates on cooling. The crystals are purified by recrystallisation.

Acidum Oleicum is obtained from oils and fats by boiling with caustic soda or lime, decomposition of the resulting soap by dilute HCl or H_2SO_4, and separation of oleic from stearic acid by refrigeration and powerful expression. The liquid portion is crude oleic acid, which is purified by treatment with oxide of lead, whereby oleate of lead is formed; this is treated with ether, which dissolves the oleate, but not the remaining stearate of lead. The ethereal solution is cleared by decantation, and mixed with hydrochloric acid; the oleic acid thereby eliminated is dissolved in the ether, which is separated from the watery liquid, and the ether recovered by distillation. If the colour is still dark, it must be decolourised by animal charcoal or fuller's-earth. The original oils may also be decomposed by the action of superheated steam. *Glycerin* is prepared as a bye-product in the same process, being present in the original solution from which the soap is removed, which is evaporated, and the residual glycerin distilled under reduced pressure. It is purified by redistillation. Two of the oleates—or rather admixtures of the true oleates with oleic acid—are official, and have already been noticed (*vide* "Oleatum Hydrargyri" and "Oleatum Zinci"). In addition to these the three official soaps—Sapo Durus, Sapo Mollis, and Sapo Animalis—are compounds of oleic and stearic acids with the alkalies. Hard soap consists mainly of oleate of sodium with a small proportion of stearate; soft soap is a similar compound of potassium; and both are officially prepared from olive oil by boiling with solution of caustic soda or potash, and subsequent removal of the soap by adding common salt to the solution, whereby it is precipitated as a curd; this is then removed, pressed, and melted, allowed to cool, and finally cut into bars or tablets in the case of hard soap. Curd soap or Sapo Animalis is made from tallow or other animal fat, and consists mostly of stearate of sodium with some oleate. The action of the alkali in this operation consists in decomposing the oils which are compounds of oleic, stearic, &c., acids with glycerin, with production of metallic salts and liberation of the alcohol glycerin; such an action, whereby alcoholic compounds of the fatty acids (ethereal salts) are decomposed with liberation of the alcohol and formation of a salt of the acid, is known as *saponification*. Other instances have already been noted (*vide Emp. Plumbi*, and imperfectly, *Linimentum Ammoniæ*, and *Linimentum Calcis*).

Acidum Salicylicum (*vide* p. 175).

Acidum Tartaricum (*vide* p. 175).

NEUTRAL PRINCIPLES.—The methods of preparation vary greatly.

Aloin.—Barbadoes aloes is extracted by means of hot water, the liquid acidified by HCl, and filtered, then evaporated to a low bulk and set aside some days to crystallise. The yellowish crystals are purified by recrystallisation.

Chrysarobinum is prepared by boiling Goa powder (araroba) with benzol, filtering, and separating the semi-crystalline matter which separates on cooling. It is dried until the odour of benzol has disappeared. Most of the chrysarobin of the shops appears to be either crude (generally very crude) araroba or *chrysophanic acid*, which is an oxidation product of chrysarobin and prepared by extracting Goa powder by means of solution of potash or soda, allowing to stand some time for oxidation to occur, and precipitating the chrysophanic acid by the addition of hydrochloric or sulphuric acid, washing and drying the precipitate.

Elaterinum.—Elaterium is exhausted with chloroform, the chloroform removed by distillation, the resulting mixture of elaterin, fat, and chlorophyll washed with ether to remove the impurities, and the elaterin finally recrystallised from chloroform. To obtain the elaterium the fresh nearly ripe fruits of the squirting cucumber are cut lengthwise, the juice lightly pressed out and strained through a hair sieve. After the sediment has deposited, the supernatant liquid is decanted, the residue poured on a linen filter, and dried on porous tiles in a warm place.

Picrotoxinum.—This and the next are by some regarded as glucosides. Picrotoxin is prepared by exhausting cocculus indicus by means of boiling alcohol, distilling off the alcohol, treating the residual oily extract with boiling water, adding acetate of lead to the filtered aqueous liquid and filtering, removing excess of lead by sulphuretted hydrogen, again filtering, evaporating the filtrate to a low bulk, and allowing to crystallise. The crystals are purified by repeated crystallisations from water.

Santoninum.—Santonica is exhausted by twice boiling with water and slaked lime and expressing the liquids. The expressed liquors are allowed to stand, syphoned off from the deposit, and evaporated to a small bulk (about three times the weight of santonica taken). Hydrochloric acid is added to the hot liquid with diligent stirring until the fluid is distinctly and permanently acid, and set aside for five days for the precipitate to subside. The lime and santonin combine to form soluble santonate of calcium, which is decomposed by the HCl, forming calcium chloride and santonin. The precipitate is collected on a paper filter, washed first with water till the washings are colourless and nearly free from acid reaction, then with a little weak ammonia, and finally with cold water till the washings are again colourless (the ammonia renders some accompanying resin and colouring matter soluble, but does not dissolve santonin). The precipitate is now dried on paper or tiles in a warm place, then mixed with a little animal charcoal, rectified spirit added, let stand half an hour, and boiled for ten minutes. The hot liquid is filtered, the charcoal washed with a small quantity of hot rectified spirit, and the filtrate set aside for two days in a dark place to crystallise. The crystals are separated, the mother-liquor concentrated to obtain more crystals, which are separated as before. These crystals are further purified by recrystallisation from boiling spirit, and finally dried on filtering paper in the dark. Santonin becomes discoloured by the action of light, hence the necessity for keeping its solutions dark.

STEAROPTENES.—Volatile oils usually consist mainly of two constituents—a liquid hydrocarbon called the *terpene* or *elæoptene*, and a solid oxygenated compound called the *stearoptene*. This oxygenated compound is generally the chief odorous principle.

Three of these stearoptenes are official in the B. P., viz. camphor, menthol, and thymol.

To prepare these stearoptenes the volatile oil is first obtained and is then usually fractionated, the earlier and more volatile fractions being rejected, whilst the fractions of higher boiling-point are submitted to *refrigeration* and subsequent pressure whilst cold of the crystalline mass which separates. If the pressed cake be now *slightly* warmed and again pressed, a further quantity of liquid oil can be expressed. This mass may then be further purified by solution in spirit and precipitation by addition of much water, or it may be crystallised from a suitable solvent, or sublimed.

On the large scale the refrigeration may be accomplished by means of *ice machines,* but for the student's purpose one of the many freezing mixtures may be employed Powdered ice or snow and salt is the best for general use ; sulphate of soda and hydrochloric acid may also be employed. The vessel containing the oil must be deeply immersed in the freezing mixture, and moved about from time to time to mix the ice and salt. *Thymol* is also obtained by treating the oil of thyme or ajowan with alcoholic potash, distilling off the alcohol, decomposing the potassium compound with HCl, and purifying by crystallisation from spirit or benzin.

Questions on Chapter XXI.

1. Define glucoside ; alkaloid ; stearoptene.
2. Name three official drugs which owe their activity wholly or mainly to the presence of each of the following : (1) alkaloid ; (2) glucoside ; (3) resin ; (4) tannin ; (5) neutral principle.
3. Give a list of the official alkaloids and alkaloidal salts, with the plants from which they are obtained.
4. Describe the preparation of atropine or aconitine, and quinine or morphine, with notes explanatory of the various stages of the operations.
5. How are the following separations effected ?—Quinine from cinchonine, strychnine from brucine, morphine from codeine, and cocaine from other coca bases.
6. Describe the preparation of tannic acid.
7. Describe two general methods for the preparation of resins.
8. Explain the use of the following : lime in preparation of santonine, ammonia in ditto, lead acetate for preparation of salicine, calcium chloride for morphine.
9. Describe a method for the preparation of stearoptenes.

CHAPTER XXII

CARBOHYDRATES, THEIR DERIVATIVES AND PREPARATIONS

Carbohydrates.—This term is applied to a widely distributed class of principles which consist of carbon, combined with hydrogen and oxygen in the proportions to form water. The molecule usually contains six, twelve, or some other multiple of six atoms of carbon, but artificial sugars have been produced, containing seven, eight, or nine atoms of carbon. The sugars, starches, gums, and cellulose are all more or less pure carbohydrates. These substances are either aldehydes or ketones, or are nearly allied to them in their chemical relationships. Carbohydrates cannot be considered as *active* principles, as they are almost universally present in plants. The official carbohydrates are starch, lactose or milk-sugar, sucrose, *i. e.* cane or beet sugar, cotton wool, which is nearly pure cellulose, acacia, and tragacanth.

Starch is officially ordered to be prepared from wheat, maize, or rice, although many other plants furnish it in abundance, notably the potato, tapioca, maranta (arrowroot), and tous-les-mois plants. Among official drugs containing abundance of starch may be mentioned pearl barley (*Hordeum decorticatum*), calumba, colchicum corm, ipecacuanha, and liquorice. The student may imitate the process of manufacture by scraping a potato into a pulpy condition, enclosing the pulp in muslin, and washing it with a stream of water; the muslin retains the fibrous matter, but permits of the passage of starch granules along with the water, from which it is deposited by settling; the water is poured off, the starch washed once, and then dried at a low temperature. The apparatus shown in Fig. 13 is very convenient for this purpose.

Starch is used to harden the suppositories of morphine and soap, and tannic acid and soap; as a diluent of compound tragacanth powder; and for the glycerine and mucilage, the last of which is a bland vehicle for all the enemata except that of asafœtida.

Sugar is obtained in the crude condition by evaporation of the juice of the sugar-cane and crystallisation. The dark-coloured sugar so obtained is dissolved in water, filtered through animal charcoal to decolourise it, and evaporated *in vacuo* to a strong syrup, then run out into conical moulds, when it crystallises in hard masses known as loaf sugar or lump sugar.

Sugar is used for many other preparations besides the syrups; it is a diluent and sweetening agent in several compound powders and lozenges; it is used to sweeten mixtures of guaiacum and brandy, citro-tartrate of sodium, and some other granular effervescent compounds, and the elixirs; it acts as a preservative in the confections of roses, hops, and senna, saccharated carbonate of iron, compound iron mixture, and pill of iodide of iron; assists the solution of lime in saccharated lime water, and prevents deposition of resinous matter during evaporation of liquid extract of sarsaparilla.

Sugar of milk.—This is obtained from milk by curdling by means of a little acid, and filtering off the whey; neutralising with lime, evaporating to a low bulk, and setting aside to crystallise.

Sugar of milk is used as a diluent of strong drugs in powders, pills, &c., *e. g.* Pulv. Elaterini comp., and is employed to prevent the "caking" of dried extract of euonymus by absorption of moisture from the air.

Gossypium, or cotton wool, the official form of cellulose, consists of the hairs of the seeds of several species of *Gossypium* (cotton plant), which are purified by treatment with weak acid and alkaline liquids to remove fatty matter and other impurities.

Among the products obtained by treatment of the carbohydrates are the following:—Alcohol, acetic and lactic acids, and pyroxylin.

Alcohol is prepared by fermentation of sugars through the agency of the yeast plant (*Saccharomyces cerevisiæ*). All kinds of sugar are not capable of fermentation; milk-sugar, for example, is not. The student should prepare a solution of half a pound of cane-sugar in two pints of water. The liquid is raised to a temperature of 21° C., introduced into a Winchester quart bottle provided with a cork and bent glass tube, leading into a small bottle of lime water. A small piece of yeast is added (half an ounce), and the whole put in a warm place at about 21°—27° C. Very soon bubbles of gas will be evolved, and the lime water will become cloudy owing to the production of chalk. The following equation represents the reaction:

$$C_{12}H_{22}O_{11} + H_2O = 2C_6H_{12}O_6.$$
$$C_6H_{12}O_6 = 2C_2H_5HO + 2CO_2.$$

The sucrose is first converted into the hexatomic sugar glucose, which, being fermentable, is decomposed, forming alcohol and carbonic anhydride. As a matter of fact some minor changes occur in the reaction, whereby glycerine, acetic and succinic acids, and some other substances are produced. When action ceases the liquid is distilled until about one half has collected; this is rendered neutral by lime or soda, and one half again distilled, when a somewhat diluted spirit is obtained.

Acetic acid results from the action of another ferment (*Mycoderma aceti*) upon weak alcoholic liquids, such as beer or the poorer wines; the dilute acid so produced is called malt or wine vinegar. It is also obtained by the destructive distillation of wood, which consists chiefly of cellulose, lignin, and other carbohydrates.

Lactic acid is yet another result of fermentation; it is obtained from sugar by solution in water, addition of a small proportion of cheese, and some chalk to neutralise the acid formed, and setting aside at a temperature of 26° C. or rather higher for about three weeks. Lactate of calcium is formed; the crystals are removed, dissolved in water, cautiously decomposed by sulphuric acid, alcohol added, the solution filtered from calcium sulphate, and the filtrate evaporated to a syrup.

Pyroxylin, or dinitro-cellulose, is prepared as follows:

"Take of—

Cotton wool	. . . . .	1 part.
Sulphuric acid, Nitric acid,	of each . . .	5 fl. parts.

"Mix the acids in a porcelain mortar, immerse the cotton in the mixture, and stir it for three minutes with a glass rod, until it is thoroughly wetted by the acids. Transfer the cotton to a vessel containing water, stir well, decant the liquid, and continue to wash by affusion, agitation, and decantation until the washings cease to give a precipitate with chloride of barium. Drain the product on filtering paper, and dry in a water-bath."—B. P.

It is important that the time should be adhered to; if it be exceeded *tri*nitro-cellulose, gun-cotton, is likely to be formed, which is not soluble in a mixture of rectified spirit 1 fl. part, ether 3 fl. parts. The H_2SO_4 is used to render the HNO_3 stronger by combining with the water which is present in the B. P. acid. Nitric acid of sp. gr. 1·42 (B. P.) does not act upon cellulose in the same way as the stronger acid. The washing till free from acid is most important, for if free acid be left in the cotton it is liable to spontaneous decomposition during or after drying.

The following equation represents the nature of the reaction:

$$C_6H_{10}O_5 + 2HNO_3 = C_6H_8(NO_2)_2O_5 + 2OH_2.$$

Surgical Dressings.—Cotton (or sometimes linen, which is the fibre from the bast of the flax plant) also forms the basis of the antiseptic wools, gauzes, &c. Since the introduction of the antiseptic system of surgery the demand for this class of preparations has increased, until at the present time there is quite a number of medicated gauzes, cottons, lints, &c. They consist of a basis of absorbent cotton wool, lint, gauze, or gauze tissue impregnated with an approximately uniform quantity of the antiseptic drug.

The materials used must be thoroughly absorbent, to attain which it is necessary that all fat should have been removed by treatment with suitable solvents. Absorbent gauze (a kind of muslin) should have at least thirty threads each way in a square inch, and should weigh about 10 drachms to the square yard. Gauze tissue consists of a layer of absorbent wool about 1¼ inch thick between two pieces of gauze.

Three principal methods of preparation are adopted:

1. *By saturation.*—Example: sublimated cotton 1 per cent. Dissolve 70 grains of mercuric chloride and 70 grains of ammonium chloride in 3 pints of distilled water, and distribute this as evenly as possible over one pound of cotton wool. Introduce into a press and apply pressure till the liquid begins to flow, then rearrange the wool in the press, adding the expressed liquid, repeat the pressure, and continue alternate addition of liquid and pressure until the wool is uniformly saturated. Now open out the wool and dry in a warm room. If no suitable press be at hand more liquid will be needed, and the hands must be used to squeeze the wool. In this and all similar operations the most scrupulous cleanliness must be observed, the hands being occasionally rinsed with an antiseptic liquid. Almost all dressings may be made by this method, although water cannot be used as the solvent in most cases; *e. g.* iodoform is best dissolved in light petroleum or a mixture of ether and alcohol, which not only act as solvents

but by their very rapid evaporation allow the wool, &c., to dry without undue loss of the volatile medicament. For thymol gauze a mixture of spermaceti and resin is commonly employed, the gauze being dipped into the melted mixture and pressed in a hot press. Carbolic acid and eucalyptus gauzes are similarly prepared, using equal parts of resin and hard paraffin.

2. *By spraying.*—This method may also be almost universally employed, but uniformity of product is less perfect. The solution of drug in alcohol, ether, or other volatile solvent is contained in a bottle, and by means of an ordinary spray producer is distributed as evenly as possible over the wool, &c.

3. *By sublimation.*—This method may be used for iodised cotton and some few other dressings. Take 4 oz. of dried absorbent wool, and distribute throughout it 87½ grains of powdered iodine. Place the mixture in a large wide-mouthed flask, immersed in boiling water for a few minutes to expel some of the air, then close it and tie down the stopper. The flask is then heated to near 100° C. in the water for two hours, when the iodine will be evenly distributed throughout the cotton, which will contain 5 per cent.

The following are the most important surgical dressings, none of which are official, but the student should prepare two or three.

Name of dressing.	Strength.	Solvent.	Remarks.
Sublimated wool	½ to 1%	Spirit and water or distilled water	Martindale recommends an equal weight of glycerine in the solution.
„ gauze			
„ „ tissue			
„ lint			
Sal-alembroth wool	2% ammonio-merc. chloride	Distilled water	Coloured pale blue with methylene blue.
„ gauze	1% do.		
„ „ tissue	2% do.		
Iodoform wool	5%, 10%, &c.	Petroleum ether or ether and spirit	—
„ gauze	10%, 20%, &c.		
„ „ tissue	Do.		
„ lint	10%, &c.		
Iodised wool	5% iodine	—	By sublimation.
Carbolic wool	6%	Spirit	Use also 5% of glycerine to prevent loss of carbolic acid.
„ gauze	4%	Hard paraffin 1, resin 1	—
Thymol gauze	1%	Spermaceti 10, resin 1	—
Eucalyptus gauze	5% oil	Hard paraffin 1, resin 1	—
„ wool	Do.	Spirit	Use also 5% of glycerine to prevent evaporation of oil.
Boric acid wool	About 50%	Boiling water	By soaking in a hot saturated solution of boric acid, coloured with cochineal.
„ lint	Do.		
Zinc-mercury cyanide gauze	2 to 3%	Suspended in water	Coloured with ammoniacal hæmatoxylin.

Antiseptic Catgut and Silk.—For surgical sutures, catgut or silk, rendered antiseptic by carbolic acid or bichloride of mercury, is employed. These may be conveniently mentioned here. For carbolic catgut, a solution of carbolic acid in olive oil, 1 to 5, is used, the immersion being prolonged to two months or more. For silk the acid is dissolved in 9 parts of melted beeswax, the silk thoroughly steeped in it, and the superfluous wax removed by drawing the threads through a cloth. For sublimated gut, first macerate in ether for twenty-four hours to remove fat, then transfer to a 1 in 1000 mercuric chloride solution in water 4 parts, alcohol 1 part, for half an hour, cut into pieces three or six feet long, dry, and macerate in oil of juniper for ten days. To use, wipe off adhering oil with an antiseptic towel, and immerse in the mercuric chloride solution. See also 'P. J.,' xxi, p. 960.

Appendix to Chapter XXII.

Pepsin.—This is not a carbohydrate, but a ferment which, in the presence of dilute HCl, has the power of dissolving coagulated albuminous substances; the B. P. directions for its preparation are:—The stomach of the recently killed pig, sheep, or calf having been cut open and laid on a board with the inner surface upwards, any adhering portions of food, dirt, or other impurity, are to be removed, and the exposed surface slightly and rapidly washed with a little cold water; the cleansed mucous membrane is then to be scraped with a blunt knife or other suitable instrument, with some pressure, and the viscid pulp thus obtained is to be immediately spread over the surface of glass or glazed earthenware and quickly dried at a temperature not exceeding 100° F. (37·8° C.). The dried residue is to be reduced to powder and preserved in a stoppered bottle.

Questions on Chapter XXII.

1. What is a carbohydrate?
2. Name the official carbohydrates with sources.
3. Describe the preparation of starch.
4. How is pyroxylin prepared? Give one important test of purity.
5. Describe the preparation of alcohol from sugar.
6. Describe the preparation of a surgical wool by sublimation.
7. What conditions should surgical gauze fulfil? What is absorbent gauze tissue?
8. Describe the preparation of iodoform wool.
9. What solvents are used for preparing carbolic gauze, thymol gauze, and eucalyptus gauze?

CHAPTER XXIII

STANDARDISED PREPARATIONS

We have already seen that most vegetable drugs owe their activity to the presence of one or more definite active principles, commonly alkaloids, glucosides, or resins. These active principles are associated in the plant with various vegetable acids, tannins, gums, saccharine and other substances.

It has been found, as might indeed have been fully expected, that the same plants growing in different countries, or under different conditions, do not contain the same proportions of these active substances; and, as was not so readily foreseen, that even when plants are grown in the same country, and as nearly as possible under the same conditions, yet their produce varies greatly in potency in different years. Moreover the season of the year at which the drugs are collected has a great influence on their strength. Under these circumstances it is not to be wondered at that science has been brought to bear upon the important question of the isolation of these active principles in a state of purity, and they have been largely used in medicine. More recently, however, attempts have been made to obtain *galenical* preparations, which shall contain a certain definite proportion of these active principles, without resorting to their previous isolation. This has been carried out by the application of methods of analysis to a portion of the galenical preparations, whereby the proportion of the active principle is ascertained, and the whole is then adjusted (by the addition of menstruum or crude drug, or some other means) to a certain "standard" of strength. On this account these preparations are called *standardised*. The standards so adopted should be based upon the analysis of a considerable number of preparations, made from different samples of the crude drug of good quality.

As a typical example of this kind of work the student should refer to a series of papers by Messrs Dunstan and Short, in the 'Pharm. Journ.' (3), vol. xiii, pp. 665, 1053; xiv, pp. 292, 441, 443, 621, and 653.

Standardised preparations form an important feature of the British Pharmacopœia, in which the following are official:*— Acidum Hydrocyanicum, Aqua Laurocerasi, Spiritus Ætheris Nitrosi, Extractum Cinchonæ Liquidum, Extractum Nucis Vomicæ, Extractum Opii, Extractum Opii Liquidum, and indirectly and more or less imperfectly Tinctura Nucis Vomicæ, Tinctura Opii, and Vinum Opii.

One of the preparations of opium and all the other official examples should be prepared by the student.

Acidum Hydrocyanicum Dilutum.

The official directions are as follows:

Take of—

Ferrocyanide of potassium . . .	2¼ ounces.
Sulphuric acid	1 fl. ounce.
Distilled water	30 fl. ounces, or a sufficiency.

Dissolve the ferrocyanide of potassium in ten ounces of the water, then add the sulphuric acid, previously diluted with four ounces of the water and cooled. Put the solution into a flask or other suitable apparatus of glass or earthenware, to which are attached a condenser and a receiver arranged for distillation; and having put 8 ounces of distilled water into the receiver, and provided efficient means for keeping the condenser and receiver cold, apply heat to the flask, until by slow distillation the liquid in the receiver is increased to 17 fl. ounces. Add to this 3 ounces of distilled water, or as much as may be sufficient to bring the acid to the required strength, so that 100 grains (or 110 minims) of it, precipitated with a solution of nitrate of silver, and the precipitate thoroughly washed and dried, shall yield 10 grains of dry cyanide of silver.

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* Such preparations as the common dilute acids, solutions of potash, arsenic, &c., are not included here, as being of a different character altogether, the starting-points being definite chemicals, and, therefore, if correctly prepared from the B. P. chemicals they will produce B. P. preparations.

Diluted hydrocyanic acid should be kept in well-corked bottles, tied over with impervious tissue. The bottles should be inverted when not in use, and be kept in a dark place.

The following equation expresses the reaction which occurs :

$$2K_4FeC_6N_6 + 6H_2SO_4 = 6KHSO_4 + FeK_2FeC_6N_6 + 6HCN.$$

During the distillation the contents of the retort are much given to "bumping"—that is, sudden evolutions of vapour, which may be so violent as to fracture the retort. Various means have been suggested to prevent this : perhaps one of the best is to introduce a number of capillary glass tubes into the liquid ; or a single piece of rather wide glass tubing, the upper end of which is closed by fusion, may be passed through the cork of the retort, the lower and open end terminating just above the bottom of the liquid in the retort.

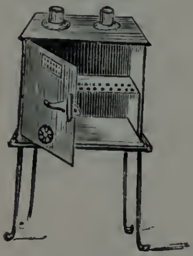
The end of the condenser tube should be connected with a glass tube or an adapter, the other end of which dips below the surface of the water in the flask. As soon as the distillation is completed, the strength of the acid may be determined either by the B. P. gravimetric test, or more conveniently by volumetric analysis.

Gravimetric analysis is the operation of determining the composition of a substance by the actual weighing of its constituents after separation, either in the free state or as compounds of known composition.

Volumetric analysis, on the contrary, consists in the determination of the quantity of a substance by means of a solution of some other substance of known strength, which exerts a definite chemical action upon the substance sought.

In the case under consideration the gravimetric test is carried out as follows :—Some of the distillate is introduced into a weighing bottle, which consists of a light-stoppered bottle ; the bottle and contents are now accurately weighed. Into a small beaker is put a solution of about 12 or 15 grains of nitrate of silver in 2 fl. ounces of water, together with about 15 minims of nitric acid. Some of the distillate (about 80 to 90 minims) is now carefully poured from the weighing bottle into the beaker, the whole stirred together, and the weighing bottle and contents again weighed. The precipitate which is formed is allowed to subside in a dark place, and meanwhile a Swedish filter-paper has been dried in the air or water oven at 100° C. The dried filter is folded, placed in coupled watch-glasses—*i. e.* two watch-glasses with accurately fitting ground edges, held together by a metal clip,—and accurately weighed. The filter is now placed in a funnel, damped with distilled water, and the nearly clear liquid passed first through the filter; the silver cyanide is washed once by decantation with distilled water, then collected on the filter and washed till the filtrate is perfectly free from acidity; it is then dried at 100° C. in the air or water oven, the former of which is shown in Fig. 68, and again weighed in the watch-glasses. The difference in weight will give the amount of silver cyanide from the quantity of distillate taken. From these data the amount of water required to dilute the whole to the official strength is readily calculated. An example will make this clear.

Fig. 68.



Weight of distillate = 16·95 ounces = 7418 grains.

Weight of weighing bottle and distillate .	287·82	grains.
„ „ „ alone . . .	202·62	„
„ distillate taken	85·20	„

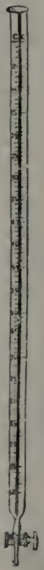
Weight of silver cyanide, glasses, clip, and filter	178·97	grains.
„ glasses, clip, and filter . . .	169·02	„
„ silver cyanide	9·95	„

That is . 85·20 grains of distillate yield 9·95 grains of AgCN.
Therefore every 85·93 „ „ „ 10·0 „ „
Wherefore to „ 89·93 „ must be added (100·00 − 85·93) = 14·07 grains of water, that is, to the remaining 7333 grains of distillate 1200 grains of water must be added to bring the whole to the required standard.

Following the B. P., the above example is given in English weights, although the author strongly recommends the student to acquire the habit of working under the metric system.

For the volumetric test a solution of silver nitrate of known strength is required; this is known as the *standard* solution. It is made by dissolving 17 grammes of silver nitrate, accurately weighed, in sufficient distilled water to produce 1 litre. The molecular weight of $AgNO_3$ is 170, consequently this solution contains one tenth of a molecular weight (in grammes) in the litre, and is therefore called a *decinormal solution*, a *normal* solution being one which contains one molecular weight in grammes in the litre. Some of this solution is introduced into a burette (Fig. 69), which consists of a long glass tube, accurately divided into equal parts, drawn out below, and provided with a perforated glass tap, or an india-rubber tube with clip. The most serviceable instrument is one containing 50 c.c. divided into tenths of a c.c. The tap having been turned so as to fill the nozzle with the solution, the level of the liquid is adjusted to the mark 0.

Fig. 69.



Into a light stoppered flask is introduced about 30 c.c. of water and the whole weighed, then 5 c.c. of the distillate and again weighed. Two drops of solution of litmus are added, and then soda solution in quantity sufficient to produce a distinct alkaline reaction. This is necessary because the reaction is not perfect except in alkaline solution, and more soda must be added during the test if the colour should return to red. The standard solution is now added, freely at first, so long as the opalescence produced is readily dissolved on agitation, but more cautiously afterwards, and drop by drop towards the end of the reaction. The reaction is complete when the liquid remains distinctly turbid on agitation and standing for a minute or two. The amount of standard solution used is then read off on the scale, and from this the real HCN in the quantity taken may be calculated.

The addition of silver nitrate to the sodium cyanide produces silver cyanide and sodium nitrate; but the former of these, although insoluble in water, is soluble in solution of sodium cyanide, forming a double salt; hence the rapid disappearance of the opalescence at first produced. When exactly one half of the cyanogen is converted into silver salt the liquid is still clear, but the addition of a single drop of silver solution now causes a precipitate, owing to the production of an excess of silver cyanide:

$$2NaCN + AgNO_3 = AgCN,NaCN + NaNO_3 \text{ (soluble).}$$
$$2NaCN + 2AgNO_3 = 2AgCN + 2NaNO_3 \text{ (insoluble).}$$

Therefore, in the volumetric test, one molecule of $AgNO_3$ is equivalent to 2 molecules of NaCN or HCN, *i.e.* 170 of $AgNO_3$ = 54 of HCN; or in other words 1000 c.c. (one litre) of the standard solution containing 17 grammes of $AgNO_3$ is equal to 5·4 grammes of anhydrous hydrocyanic acid, therefore 1 c.c. of the standard solution = 0·0054* gramme HCN.

For example, 5 grammes of distillate required 24 c.c. of standard solution.
Therefore 5 „ „ = 24 × ·0054 gramme = 0·1296 gramme HCN = 2·592 per cent.

Wherefore every 2 parts by weight require 0·592 part by weight of water to produce the official diluted hydrocyanic acid.

Scheele's hydrocyanic acid is double the B. P. strength.

The acid should be carefully preserved as described, to prevent loss of strength by volatilisation and decomposition with production of a brownish insoluble matter by action of light.

Aqua Laurocerasi.—This is prepared by distillation in the same way as other medicated waters. One part of the chopped and crushed fresh leaves is introduced into a retort with 3⅛ parts of water, and 1¼ part of liquid distilled. Cherry laurel leaves contain a principle known as laurocerasin, which, under the influence of water and a ferment allied to emulsin (a constituent of almonds) also present in the leaves, is decomposed with production of hydrocyanic acid, benzoic aldehyde (oil of bitter almonds), and sugar. The benzoic aldehyde and hydrocyanic acid distil over with the water. It is to the latter that the preparation owes its efficacy. It has been found that the proportion of HCN present varies greatly with the season, leaves gathered in the winter or spring yielding far less than those gathered in summer or autumn: young leaves are commonly more powerful than old ones. The proportion required by the B. P. is 1 part of HCN in 1000; it may be estimated by the volumetric test described under hydrocyanic acid. The B. P. directs the distillate to be agitated, filtered through paper, and the strength adjusted by addition of distilled water or diluted HCN; this should not be done until the preparation has stood about a week, as it loses strength for the first few days after preparation, but then a state appears to be attained when the strength is nearly permanent.

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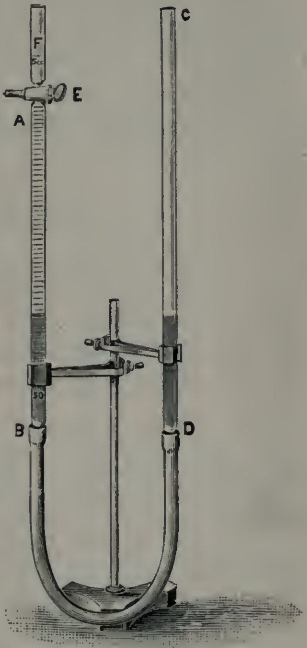
* This number, the amount which each c.c. of a volumetric solution is equivalent to, is called the *factor*.

Fig. 70.



Spiritus Ætheris Nitrosi.—This is prepared as described in Chap. XI, p. 129, and is assayed as follows:—A nitrometer (Fig. 70) is filled with strong brine, the graduated limb A B being quite full, as well as the india-rubber tube B D and part of the open tube C D. The tap E is then closed, and some of the brine removed from C D. Five c.c. of the sample of spirit is then introduced into the cup F, and cautiously run into the tube A B by turning the tap E, taking care that no air gets in at the same time. Five c.c. of a strong solution of potassium iodide is next allowed to enter, and this is followed by about 5 c.c. of dilute sulphuric acid. Effervescence immediately ensues, and the tube should be vigorously agitated at intervals by holding the tapped tube vertically and agitating the contents by a transverse motion. In this way the reaction is readily confined to the upper part of the liquid in the closed side of the nitrometer. The reaction is complete in five minutes, when the *level of the liquid in the two limbs of the nitrometer is adjusted,* and the volume of nitric oxide gas read off. The calculation of the percentage of ethyl nitrite is based upon the following equation:

$$(C_2H_5)NO_2 + KI + H_2SO_4 = (C_2H_5)OH + KHSO_4 + I + NO.$$

The B. P., however, simply requires that when freshly prepared the volume of gas liberated shall be seven times that of the spirit taken, or after keeping some time, at least five times its volume. The strength of the spirit should be adjusted by addition of rectified spirit to fulfil the former of these, which corresponds to 2·65 per cent. by weight of ethyl nitrite.

This is Allen's process, an alternative method is given; it depends upon the evolution of NO when an acidulated solution of ferrous sulphate is heated with a nitrite (*vide* 'P. J.,' xiii, p. 63, and xv, p. 101). The nitrometer method is, however, much simpler, and sufficiently accurate for all practical purposes.

Extractum Cinchonæ Liquidum.

Take of—

Red cinchona bark, in No. 60 powder	20 ounces.
Hydrochloric acid	5 fl. drachms.
Glycerine	2½ fl. ounces.
Rectified spirit, Distilled water, } of each	a sufficiency.

Mix the bark with five pints of the water to which the acid and glycerine have been added, and macerate in a covered vessel for forty-eight hours, stirring frequently; then transfer to a percolator, and when the fluid has drained out continue the percolation with water until the liquid which is passing ceases to give a precipitate on the addition of excess of solution of soda. Evaporate the percolate in a porcelain or enamelled iron basin at a temperature not exceeding 180° F. (82·2° C.) until it is reduced to twenty fl. ounces.

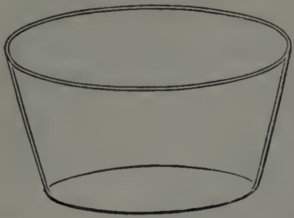
This liquid is now estimated as follows:—"2·5 c.c. are introduced into a separator with 10 c.c. of distilled water, 10 c.c. of benzolated amylic alcohol (benzol 3 vols., amylic alcohol 1 vol.), and 10 c.c. of solution of soda, and the whole thoroughly agitated, then allowed to rest until the liquids have separated. The lower layer is removed by the tap, and 10 c.c. more of distilled water introduced and agitated with the lighter liquid which contains the bulk of the alkaloids originally present in the extract; after standing to separate, this water is run off as before, the alkaloidal solution being poured into a small weighed glass or porcelain dish. The alkaline liquid which was first removed is now agitated with 10 c.c. more of benzolated amylic alcohol, separated as before, and the alcohol washed with the wash-water used before, and added to the alcohol in the dish. This is then evaporated on a water-bath until dry, and weighed; it is further heated for an hour upon the water-bath or in an air-oven (at 105°—110° C.) and again weighed, and if any loss of weight has occurred, this heating and weighing must be repeated until the weight is constant.

The weight of the dish and residue, *minus* the original weight of the dish, gives the weight of alkaloids from 2·5 c.c. of the evaporated liquid, and this multiplied by 40 gives the number of grammes from 100 c.c., which is the same as grains in 100 *fl. grains.* Having thus ascertained the strength, every fluid part of it containing 5 grains of alkaloids is brought to the measure of 85 fl. grains by evaporation, or if too strong by dilution with water; then 12·5 fl. grains of rectified spirit are to be added, and finally sufficient distilled water to produce 100 fl. grains. The finished product will then contain 5 parts of alkaloids in every 100 fl. parts.

The acid is used for the purpose of aiding the extraction of the alkaloids by converting the almost insoluble cinchotannates into hydrochlorates. The glycerine has the double effect of aiding extraction, and preventing the decomposition of the peculiar tannic acid of the bark. The percolation is very tedious, and complete exhaustion seems to be unattainable. The evaporation is best conducted *in vacuo*, as by heat much of the alkaloid is rendered insoluble, especially if the temperature approaches ebullition; hence the direction to evaporate below 82° C. It should be diligently stirred, and the weakest liquors evaporated first; when reduced to the proper volume it should be thoroughly cooled and filtered through calico, or the finished product will not mix clear with distilled water.

Estimated as described with double washing with benzolated amylic alcohol, more perfect exhaustion is attained than by the B. P. single washing. The agitation in separator should be a rapid rotatory motion, not a violent motion up and down, which latter often effects such an admixture of the liquids that no clear separation can be obtained afterwards; this remark is even more important as applied to the next assay to be described. The dish used in the operation should be a flat-bottomed one, preferably with straight or nearly straight sides and ground edges, so as to avoid the habit of "creeping up" which many solutions exhibit. Fig. 71 represents the form of glass dish which the author prefers; it possesses all the advantages of a dish with vertical sides, and will fit into the rings of a water-bath more readily.

Fig. 71.



The yield by this method is very seldom 20 fl. parts, as the official bark contains 5 to 6 per cent. of alkaloids, a portion is always left behind in the marc, and some is lost bybecoming insoluble during the process.

Extractum Nucis Vomicæ.

Take of—

Nux vomica	1 part.
Rectified spirit	4 fl. parts.
Distilled water	1 fl. part.

The seeds are split and heated to 100° C. for three hours, then reduced to fine powder, and exhausted by percolation with the mixed spirit and water. The alkaloids in the mixed percolate are now estimated as follows:

Twenty-five c.c. are evaporated almost to dryness over the water-bath, the residue rinsed into a separator with 10 c.c. of chloroform, 15 c.c. of dilute sulphuric acid, and 15 c.c. of distilled water; gently warmed and agitated. When the liquors have separated draw off the chloroform, and add to the acid liquor 10 c.c. of chloroform and solution of ammonia to a strongly alkaline reaction, again agitate and warm; when separated draw off the chloroform, and repeat the agitation with 5 c.c. of chloroform twice. Wash the chloroformic solutions, which contain the alkaloids, successively with about 8 c.c. of distilled water, and transfer them to a weighed dish, evaporate, heat the residue to 100° C. for one hour, cool and weigh; the weight multiplied by 4 gives the amount of alkaloid in 100 fl. parts. Take of the percolated liquid as much as contains 150 grains of dried alkaloids, distil off the spirit, and evaporate to 1000 grains in a tared dish, or in this proportion. The resulting extract will contain 15 per cent. of alkaloids.

It is almost impossible to grind the seeds unless first dried as described. Spirit of the official strength is the most suitable solvent; it dissolves the alkaloids very perfectly, but extracts scarcely any oil, and none of the mucilage or albumen which abound in the seeds. Rectified spirit dissolves much oil, which not only renders the extract oily, but impedes evaporation.

In the assay the washing with chloroform while acid removes some oil and colouring matter.

The alkaloids consist of strychnine and brucine; the former of these is much the more active of the two, and on this account it would perhaps be preferable if the extract were standardised to contain a definite proportion of strychnine, the brucine being allowed some range to meet the variation which naturally occurs in the proportions of the two. This separation may be effected by the method of Dunstan and Short, by which the strychnine is precipitated as ferrocyanide from an acid solution, washed, decomposed by ammonia, and the liberated strychnine dissolved in chloroform, the chloroform evaporated, and the residual strychnine weighed. For details of the process see 'P. J.' (3), xiv, p. 291.

The extract finally obtained by the B. P. process will vary in consistency according to the relative richness of the seeds in alkaloids and extractive matter; to avoid this source of inconvenience and inaccuracy the author has proposed to percolate the residual marc with a weaker spirit, consisting of a mixture of rectified spirit 3 parts, water 2 parts; thus obtaining a percolate containing a larger proportion of extractive. The two percolates are evaporated separately to a soft pilular consistence and assayed, the latter being used to dilute the former if needful. Should the first extract, however, be too weak, it will be necessary to treat it with a stronger spirit, so as to dissolve the alkaloidal salts and a portion of the extractive, filter, evaporate, and assay the extract obtained.

Tinctura Nucis Vomicæ.—This is prepared by dissolving 133 *grains* of the standard extract of nux vomica in a mixture of 4 *fl. ounces* of distilled water with sufficient S. V. R. to produce 1 *pint,* and filtering if necessary. So prepared it will contain 1 *grain* of the alkaloids in *each fl. ounce,* or 0·23 *part in* 100 *fl. parts.*

Tinctura Opii and the other opium preparations are officially prepared from opium containing 9·5 to 10·5 per cent. of morphine; they are thus indirectly standardised within small limits, but the B. P. further requires that the extract and liquid extract shall be assayed and contain "about 20 per cent." and "about 1 per cent." respectively.

The opium may be assayed as follows, which is a slight modification of the official method:

Take of—

Opium	10 grammes.
Slaked lime	5 ,,
Chloride of ammonium	2 ,,
Rectified spirit	6 c.c.
Absolute ether	30 ,,
Distilled water	98 ,,
Saturated solution of morphine in distilled water	40 ,, or q. s.

Rub the opium in a mortar with sufficient of the water to completely soften it, add the slaked lime, and rub thoroughly together, then add gradually the remainder of the water. Allow to stand, with occasional stirring, for fifteen to twenty minutes, then filter through a dry filter. Collect 70 c.c. of the filtrate and introduce it into a stoppered bottle or flask capable of holding about double the quantity, add the rectified spirit and 24 c.c. of the ether, shake together, then add the chloride of ammonium, and agitate during half an hour to promote separation of the morphine in a finely divided crystalline condition; allow to stand during one night, after which the ethereal portion is carefully poured on to a small filter wetted with ether, the remaining 6 c.c. of ether added to the contents of the bottle, agitated slightly, and poured off to wash the filter. Allow the filter to dry by evaporation of ether, then add the aqueous liquid with as much of the precipitate as possible. The bottle is rinsed out and the filter washed with several successive small portions of the saturated solution of morphine in water.* When the filtrate is colourless the filter is removed from the funnel, dried by pressure between folds of blotting-paper, and then in the air-oven, raising the temperature gradually to 100° C., which is maintained until perfectly dry. The precipitate is removed from the filter to a tared water-glass, the last portions being detached by means of a stiff camel's-hair pencil. The difference between the weight of watch-glass alone, and glass with morphine, gives the amount of morphine in 7 grammes of the opium, which divided by 0·07 gives the percentage. The purity of the resulting morphine may be judged by its colour, solubility in lime water, and freedom from ash when incinerated, or better still by titration with standard hydrochloric acid, using methyl orange as an indicator.

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* Prepared by digesting about half a gramme of pure morphine in 250 c.c. of distilled water for two days with occasional agitation. It is kept with the excess of morphine, so that the solution remains saturated under varying conditions of temperature.

The theory of this method is as follows:—Morphine is soluble in solution of lime, consequently it is obtained in the first filtrate, the lime also rendering much of the soluble matter insoluble by combining with meconic acid and liberating the alkaloids. The subsequent addition of chloride of ammonium produces calcium chloride, liberating ammonia with consequent precipitation of the morphine; the small proportion of spirit causes this precipitate to become more distinctly crystalline, whilst the ether dissolves narcotine. The use of a morphine solution for washing prevents loss of alkaloid by solution in water.

Should the opium used be found to yield too high a percentage of morphine, as is usually the case, a correspondingly smaller quantity must be employed. The extract, tincture, and other preparations may be assayed by an obvious adaptation of the above method, the spirit being first removed from the liquids by evaporation. The preparations of opium are so important that the list is reproduced here, with proportion of the drug in each.

Confectio Opii	1 in 40 nearly. Made from compound powder.
Emplastrum Opii	1 in 10.
Enema Opii	1 in 426 fl. parts nearly. Made from tincture ($\frac{1}{2}$ fl. dr. in each).
Extractum Opii	2 in 1 nearly.
,, ,, liquidum	1 in 10 fl. parts nearly. Made from extract.
Linimentum Opii	1 in $26\frac{2}{3}$ fl. parts. Made from tincture.
Pilula Ipecacuanhæ cum Scillâ	1 in 23 nearly.
,, Plumbi cum Opio	1 in 8.
,, Saponis Composita	1 in 6 nearly.
Pulvis Cretæ Aromaticus cum Opio	1 in 40.
,, Ipecacuanhæ Compositus	1 in 10.
,, Kino Compositus	1 in 20.
,, Opii ,,	1 in 10.
Suppositoria Plumbi Composita	1 in 18 nearly. (1 grain in each.)
Tinctura Camphoræ Composita	1 in 220 fl. parts nearly.
,, Opii	1 in $13\frac{1}{3}$ fl. parts.
,, ,, Ammoniata	1 in 88 fl. parts nearly.
Trochisci Opii	$\frac{1}{10}$ grain extract in each.

Unguentum Gallæ cum Opio . . 1 in $13\frac{2}{3}$ nearly.
Vinum Opii . . . . . . 1 in 10 nearly. Made from extract.

Also the alkaloids codeine and morphine.

Unofficial standardised tinctures.—There are many other drugs, the preparations of which are readily amenable to methods of standardisation, and the importance of which is evidenced by the fact that the author has met with samples of tinctures which vary to such an extent that the strongest has been as much as three times as powerful as the weakest. These discrepancies are especially noticeable in the cases of conium, belladonna, cinchona, gelsemium, and jalap. Messrs Wright and Farr have also shown this fact very clearly, and have submitted methods of assay for the tinctures of conium, aconite, jaborandi, belladonna, hyoscyamus, stramonium, colchicum, gelsemium, cinchona, and veratrum.*

The following table is a summary of the results obtained by them :

Tincture.	Official menstruum.	Proposed menstruum. Alcohol by volume.	Alkaloidal contents per cent.	Proposed alkaloidal standard.	Process recommended for preparation.
		Per cent.			
Aconite .	Rectified spirit =90% by vol.	70	·045 to ·086	—	Percolation.
Belladonna .	Proof spirit= 57% by vol.	50 or 60	·015 to ·045	·025	Macero-percolation or percolation.
Cinchona .	Proof spirit	70 or 80	·69 to 1·48	—	,,
Colchicum .	,,	50	·064 to ·119	—	Percolation.
Conium .	,,	70	·06 to ·16	·08	,,
Gelsemium .	,,	60 or 70	·020 to ·076	—	,,
Hyoscyamus	,,	50	·008 to ·015	·01	Macero-percolation or percolation.
Jaborandi .	,,	50	·040 to ·152	·10	Percolation.
Stramonium	,,	60 or 70	·020 to ·034	·025	Macero-percolation or percolation.
Veratrum .	Rectified spirit	70	·028 to ·22		Percolation.

The following methods may be employed for the most important of these :

Tincture of conium.—The tincture should be made by percolation with a mixture of rectified spirit 4 fl. parts, water 1 fl. part, the percolation being stopped when about two thirds of the full volume has passed. The strong tincture so obtained is estimated as follows :—50 c.c. are introduced into a porcelain dish with 1 c.c. of normal sulphuric acid, and the mixture evaporated to low bulk over a water-bath with constant stirring. A little distilled water is then added if necessary, and the evaporation continued until all the spirit is driven off. The solution is then poured into a separator, and the dish rinsed out with 15 c.c. chloroform and afterwards with a little distilled water. These are added to the solution in the separator, the whole well shaken and allowed to stand until the chloroform has subsided. The chloroformic layer is then drawn off, and the process repeated with a second 15 c.c. chloroform. The acid solution is now shaken up with 2 c.c. B. P. Liq. Ammoniæ, and the liberated alkaloids taken out by shaking first with 15 c.c. and afterwards with 10 c.c. chloroform. The chloroformic alkaloidal solution is washed with a little distilled water (to free it from traces of ammonia), and is then run into 10 c.c. of a saturated solution of dry hydrochloric acid gas in ether, the end of the funnel tube being allowed to dip beneath the surface of the acid ether. The solution is allowed to evaporate in a current of air, and the residue heated in a water-oven at a temperature not exceeding 90° C., until it ceases to lose weight. The weight obtained × 2 represents the percentage of alkaloids as *hydrochlorates.* The tincture is now diluted with spirit of the original strength in such proportion as to produce a tincture containing 0·09 part of hydrochlorates of the alkaloids in 100 fl. parts. See also 'P. J.,' xviii, 511, and xxi, p. 558.

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* See 'P. J.' [3], xxi, pp. 857, 957, 1037; xxii, pp. 1, 255, 469, 569; xxiii, p. 248; 'C. and D.,' ii, 1892, pp. 263, 651.

Tincture of aconite.—Prepare a strong tincture exactly as for conium, and estimate as follows:—To 50 c.c. add about ·2 grm. of tartaric acid and evaporate on a water-bath to a syrupy consistence; add now 10 c.c. of distilled water and pour into a separator, rinse the dish with 5 c.c. of ether, then again with 5 c.c. of water and finally 5 c.c. more ether, pouring the rinsings into the separator. Agitate thoroughly and draw off the watery liquid. Wash the ether once with water and draw off into a separate vessel; again wash the aqueous liquid with 5 c.c. of ether, and rinse this with the water used for washing the former ethereal liquid. Now mix the aqueous liquids, add 10 c.c. of ether, render alkaline with ammonia, and agitate thoroughly; when separated wash the ether with 10 c.c. of water and introduce it into a dish or flask; agitate the aqueous liquid with 5 c.c. more ether, and repeat with another 5 c.c., washing these with the watery washing; evaporate or distil off the ether from the alkaloids, and dry at 100° C. until it ceases to lose weight. From this result the whole may be diluted so as to contain 0·05 per cent. of alkaloids soluble in ether. See also a paper by A. H. Allen, 'P. J.,' xxii, p. 230.

Tincture of belladonna.—Use proof spirit. 100 c.c. of the strong tincture is evaporated to about 20 c.c. on the water-bath, 5 drops of dilute sulphuric acid added, the whole well stirred with about 1 gramme of silica, and filtered into a separator, washing the filter with distilled water. The silica aids filtration by absorbing the oily and resinous substance which separates. To the filtrate are added 10 c.c. of chloroform and sufficient ammonia to impart a strong alkaline reaction. After separation the alkaline liquid is washed with three more quantities of chloroform, each of 5 c.c. The mixed chloroformic solutions are washed once with ammoniated water, and then agitated with three successive 5 c.c. of acidulated water. The mixed acid solutions, after once washing with chloroform, are rendered alkaline with ammonia, and the alkaloid extracted by agitation with three successive 5 c.c. of chloroform. This chloroformic solution after washing is evaporated, and the residue dried on the water-bath and weighed. If allowed to evaporate spontaneously the alkaloid will be crystalline. The whole is now adjusted to the strength of 0·025 per cent. of alkaloids by addition of proof spirit.

Tincture of henbane and tincture of stramonium.—These tinctures may be assayed in the same way as tincture of belladonna, but the separations are usually more difficult owing to emulsification of the chloroform. Various devices for overcoming this difficulty will be learnt by experience; changes of temperature, addition of spirit to the chloroform, removal of mucilage by spirit before the separation, and the judicious use of a bent wire as a stirrer, are the most commonly useful. These last three tinctures may also be prepared with a rather weaker spirit without reducing their alkaloidal strength.

Tincture of henbane should be adjusted to 0·010 per cent. alkaloid.

Tincture of stramonium should be adjusted to 0·025 per cent. alkaloid.

Tincture of cinchona and compound tincture of cinchona.—10 c.c. of tincture, 10 c.c. of benzolated amylic alcohol, 5 c.c. of solution of soda, and 30 to 40 c.c. of distilled water are introduced into a separator, and the alkaloids removed as described for liquid extract. The benzolated solution is not evaporated, however, but is in turn agitated with four successive quantities of 5 c.c. of warm dilute hydrochloric acid. This acid solution is washed once in the separator with chloroform, then rendered alkaline with ammonia, and the alkaloids extracted with ether-chloroform (equal parts) in the usual way. The washed ether-chloroform solution is then evaporated, the residue dried at 110° C., and weighed. The simple tincture should be adjusted to contain 0·8 per cent., the compound tincture 0·5 per cent. A spirit should be used rather above the strength of proof spirit, viz. S. V. R. 3 parts, water 1 part; and the addition of 5 per cent. of glycerine assists in preventing deposition of alkaloids on keeping.

Tincture of jaborandi.—Prepare a strong tincture as above, using proof spirit, or a mixture of S. V. R. 5 fl. parts, water 4 fl. parts, and estimate as described by Farr and Wright, 'P. J. (3), xxii, p. 2.

Tincture of jalap.—Make a strong tincture as described under tincture of conium, and estimate as follows:—25 c.c. are evaporated nearly to dryness in a weighed dish on the water-bath, then washed three times with 10 c.c. of distilled water at about 40° C., the water being passed through a small filter. Any small particles of resin are washed from the filter by means of rectified spirit, and the solution added to the contents of the dish, evaporated, and dried. The weight of the dish and contents − the tare of dish × 4 gives the percentage of resin in the tincture, which is then adjusted by addition of menstruum to contain 1·25 per cent. If proof spirit be used for the tincture as ordered in the B. P., it will be necessary to dissolve the resin out of the evaporated extract by means of rectified spirit, and then to treat this solution as described.

Questions on Chapter XXIII.

1. Explain the necessity for standardised preparations.
2. Define "volumetric analysis," "normal solution."
3. Describe the preparation and estimation of cherry-laurel water.

4. Name the standardised preparations of the 'British Pharmacopœia,' with the proportion of active principles in each.

5. Give the equation for the preparation of hydrocyanic acid.

6. Describe the preparation of liquid extract of cinchona, with reasons for use of acid and glycerine, and temperature employed.

7. Describe the preparation of extract of nux vomica, giving the reasons for use of spirit of special strength.

8. Describe the assay of opium.

9. Give a method for assaying tincture of belladonna.

10. What is the most satisfactory menstruum to use for the following tinctures?—Belladonna, conium, stramonium, jaborandi, aconite, jalap, henbane, cinchona, and conium.

CHAPTER XXIV

COATED PILLS, CAPSULES, CACHETS, PASTILLES, MEDICATED GELATINES, SALVE MULLS, PLASTER MULLS, TABLETS, LAMELS

THE various operations described in this chapter are of a special character, and cannot conveniently be classed under any of the sections hitherto considered. These preparations are usually produced on the manufacturing scale by wholesale firms, but a knowledge of the processes by which they may be made will frequently prove of value to the pharmacist.

Pill Coating.—For some years past it has been customary to cover pills with a tasteless coating, so as to make them more pleasant to the taste and sight, and also in some cases to prevent them from being dissolved until they have entered the lower bowel. The substances commonly used for pill coating are sugar, French chalk, gelatine, various resinous varnishes such as tolu and sandarac, gold or silver leaf, and keratine. If pills are intended to be coated they must be made hard, and very little dusting powder used; they should also be allowed to dry by exposure to the air for a few days, unless the object of coating be to retain some volatile or easily oxidised ingredient.

Varnish Coating.—The simplest form of coating is the varnish. One ounce of sandarac is dissolved in 2 fl. ounces of alcohol 65 o. p., and strained through muslin. The pills are put into a covered jar such as are used for ointments, some of the varnish poured over them, and rapidly revolved a few times, then turned out on to a porcelain slab or dish; the pills are then separated by a glass rod (dipped in alcohol to prevent sticking) and occasionally turned over. After exposure for from twelve to twenty-four hours the pills may be put into boxes or bottles. Other varnishes are used in a similar manner.

Silver or Gold Coating.—Coating with gold or silver leaf is carried out in a round-bottomed covered jar, similarly. The pills are first revolved rapidly in a covered jar with a small quantity of diluted mucilage (1 part of B. P. mucilage to 2 parts water) so as to make them slightly sticky, then turned out into a round-bottomed jar containing the silver or gold leaf, of which two leaves are needed for every dozen five-grain pills; they are rotated in this until invested with a complete coating, then the superfluous leaf blown away, and the pills polished by further rotation. Silver leaf must never be used for pills containing asafœtida, or they will be blackened by production of silver sulphide.

Gelatine Coating.—Gelatine is a very advantageous coating; it does not largely increase the bulk of the pills, and is readily soluble in the body.

For gelatine coating, roll the pills from a good stiff mass, allow from one to seven days' exposure to dry, thinly coat with a varnish half the usual strength; when dry put the pills on needles, the blunt ends of which are fixed into a wooden cylinder which can be revolved by means of a handle. The cylinder carries several (6 to 8) rows of such needles. When revolved, the pills dip into a bath containing the gelatine solution, kept at a temperature of 60° to 70° C. The pills only should dip into the solution, not the needles, or the result will be "tailed" pills. When all have been dipped, the bath is removed and the cylinder slowly revolved until the gelatine solidifies; if revolved too quickly the gelatine solution will be forced towards one end of the pills, and be unevenly distributed. Take off the pills when they are free from stickiness, but before they are *hard*, and put into trays to harden. Finally cover the pin-hole by a a small drop of the gelatine solution, or by dipping each pill into the solution for about one fourth its depth. The gelatine solution is made as follows:

Gelatine, 2 parts; distilled water, 12 parts. Dissolve by a gentle heat, add white of egg, ½ part, and raise to the boiling-point; strain through flannel, and add glycerine

$\frac{1}{2}$ part, mucilage of acacia (pale) 1 fl. part, and boric acid $\frac{1}{20}$ part.

It is important that glycerine should be used very sparingly or not at all in making up the mass, or the coating will gradually become soft.

Pearl or Chalk Coating.—For pearl coating it is even more imperative that the pills should be hard and dry. They should first receive a thin coating of varnish, or a thick one if much oil be present, and allowed to dry. For the actual coating, two covered jars, one round tin of similar size and shape to the jars, and a flannel or paraffined canvas bag will be needed. The last should be about a yard long and six or eight inches wide, the ends being open.

The pills are placed in one of the covered pots, and moistened with a mixture of mucilage of acacia, syrup, and water in equal parts, by rapid revolution. A quantity of French chalk is placed in the tin, the pills thrown into it, jolted forcibly to separate any which may be adhering, and then steadily revolved during two or three minutes. They are turned out, loose chalk separated by lightly rolling in a sieve, and partially polished by a few revolutions in the other covered pot. They are allowed about an hour to dry, and the operation repeated once or twice till the coating is thick enough, the last time being polished for longer time to make quite smooth. Finally they are transferred to the bag, and one end being held in each hand they are rolled rapidly from end to end in a warm, dry place till a good polish appears.

Sugar Coating.—First brush and sift to remove all adherent dusting powder, and introduce into a round-bottomed revolving pan, heated by steam, like a miniature confectioner's revolving pan. When warm, add a syrup containing 1$\frac{1}{2}$ of sugar to 1 of water with some powdered starch, and keep revolving, at the same time rousing up with the hand until dry, then revolve without disturbing them. Repeat this operation till white and even. Take out the pills, and when dry remove dust by shaking in sieve, and let stand over night. Clean the pan with water, and treat again with syrup containing 2 of sugar to 1 of water, just the same. Then wash and dry the pan, and treat with a weak syrup, 1 of sugar to 1 of water, in the same way, but do not continue the rousing up with hand after the pills are *nearly* dry. Now separate dust as before, and polish in a paraffined canvas bag as for pearl coating. See 'P. J.' [3], xv, p. 1070.

Keratin Coating.—Keratin coating is employed for such pills as are intended to pass through the stomach and into the small intestine before being dissolved; this is desirable when the medicines act as irritants to the stomach, or impair digestion by precipitating the pepsin, or are themselves rendered inactive by the acid gastric juice, and also in the case of anthelmintics, *i. e.* medicines for intestinal worms.

Keratin is prepared in alkaline and also in acid solution, the alkaline solution being employed for alkaline medicines, the acid solution in other cases.

Parings of horn are digested with a liquid consisting of pepsin 1 part, hydrochloric acid 1 part, and water 11 parts, so long as anything is removed. The residue is then dissolved in ammonia by prolonged maceration (several weeks) and the solution evaporated.

Another method is to digest the quills of birds' feathers in warm water for ten hours, then in a mixture of equal parts of alcohol and ether for eight days, to remove fat and cholesterin. The residue is boiled in glacial acetic acid for twenty-four to thirty-six hours under a return condenser. The thick liquid is then filtered through glass-wool and evaporated to dryness.

The alkaline solution is made by dissolving 7 parts of this dried keratin in 100 parts of a mixture of equal volumes of solution of ammonia and alcohol; the acid solution is of the same strength in acetic acid.

A fatty excipient should be used for the pill-mass and vegetable dusting powders avoided, the pills being dipped in melted oil of theobroma before coating. The coating may be carried out as with gelatine or varnish, but requires to be repeated several times.

Pill Machinery.—On the manufacturing scale, the various operations of pill-making, rolling, coating, &c., are carried out in specially constructed machinery. It will be necessary here to only give a bare outline of such processes.

The powders having been mixed by trituration or other means, they are transferred together with the excipient to the kneading machine, a convenient form of which is that known as the "universal," it consists essentially of two strong iron "blades" which revolve in an iron box; the construction is shown in Fig. 72. When kneaded, it is put into the "piping" press, which is a press the box of which has a perforated bottom; when pressure is applied to the mass it is forced through the holes in the form of cylindrical rods, which are cut into convenient lengths; of course the size of the holes varies for pills of different sizes. These cylinders are then cut by the "cutting" machine, which consists of two metal cylinders, grooved all round with hemispherical grooves, like the cutting part of a hand pill machine. These cylinders revolve in opposite directions very close together, so that the pipe of pill-mass is cut into more or less perfect pills. From this the pills are removed to be finished, which may be done by a large hand-finisher of the usual kind or by a special machine.

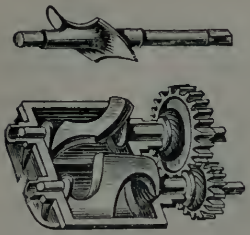


FIG. 72.—Showing principle of "Universal" Kneading Machine.

For coating the pills, apparatus similar to that already described may be used, but on a larger scale; and the final polishing is commonly carried out in a metal revolving pan without any powder.

Capsules.—Many nauseous or oily drugs may be conveniently taken in the form of capsules, which consist of thin cells constructed of gelatine with just sufficient glycerine to prevent them from becoming hard. Only such drugs as do not dissolve or act chemically upon the gelatine can be given in this form, hence it is restricted principally to oils, balsams, and some dry powders. The following is a method of preparation taken from the Pharmacopée Française:

"Take of

Colourless gelatine	25 parts.
Glycerine	10 "
Sugar	8 "
Distilled water, about	45 "

"Dissolve in a water-bath. Plunge into this solution small olive-shaped mouldings of tinned iron, slightly oiled, and fixed on a tray by means of a thin wire. Withdraw the tray after a few seconds, and give it a circular movement until the gelatinous matter has slightly cooled. When dry enough quickly pull off the capsules and trim with scissors. In order to fill the capsules place them on supports—holes pierced in a wooden board—and introduce the liquid by means of a thin pipette. Close each capsule with a drop of warm gelatinous solution, and to complete the union plunge the capsules once more into the solution to about a fourth of their length. Dry in the open air or a moderately heated stove."

Solids are usually put into capsules with one end open, having a lid which fits over the open end, the form being a cylinder with rounded ends.

Very volatile liquids, such as nitrite of amyl, iodide of ethyl, &c., are enclosed in very thin glass bulbs having a capillary aperture, which is closed by fusion after filling. For use the capsule is crushed in a handkerchief and the vapour inhaled.

Cachets.—Cachets, or wafer capsules, are small receptacles of wafer-paper in which nauseous drugs may be administered with comfort. To prepare the capsules procure wafers in sheets such as are used by fancy cake bakers. Cut them into circular pieces by means of a hollow punch. One of these is slightly moistened by placing it between wet muslin cloths, removed, and inserted between two tin plates which have been prepared of the desired shape of the finished disc, which is usually somewhat saucer-shaped; after pressing the plates together the wafer will have received the desired impression, and may be dried at a gentle heat. Some kinds of wafer-paper do not require moistening, but simple pressure between heated plates. The medicine having been weighed and placed in one of these discs, the rim of another is moistened with water or very weak mucilage, and the two joined by pressure at the rim. Small presses are made which are very convenient for closing the cachets. A good form is that shown in Figs. 73, 74, and 75.

Fig. 73.

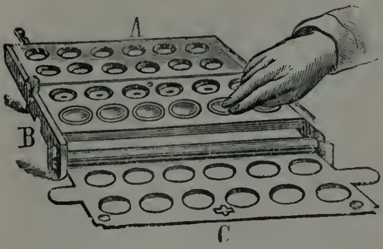


Fig. 74.

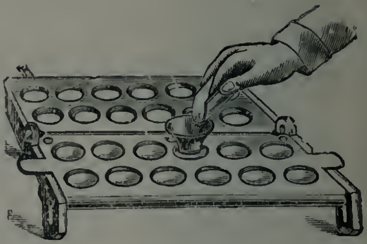
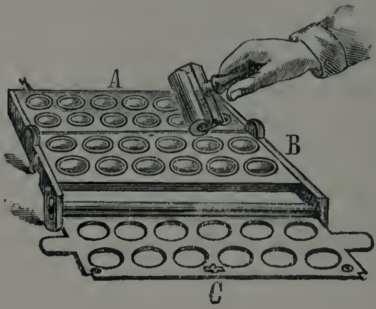


Fig. 75.



The cachets are pressed with the fingers into the inner spaces of plates A and B (Fig. 73).

Plate C is then laid on to B, and the powder placed in the cachets—large doses by means of the funnel (Fig. 74)—and pressed down. When all the cachets are filled, plate C is returned to its former position, and the damping roller (not too wet) passed over the cachets in plate A only (Fig. 75), which plate is then closed over B. A slight pressure closes all the cachets, which, on opening the apparatus, are found adhering to plate A, complete and ready for use. They are pushed out with the fingers.

Pastils.—Another pleasant form for the administration of medicines is the pastil, pastille, or jujube. The active drug is mixed or dissolved in a firm jelly composed of gelatine and glycerine suitably flavoured, and sometimes coloured.

The usual base is the glyco-gelatine of the Throat Hospital Pharmacopœia, which is made as follows:

Gelatine, in shreds	1 part.
Glycerine	2½ parts.
Orange-flower water	2½ ,,
Ammoniacal solution of carmine . .	q. s.

Soak the gelatine in the orange-flower water for two hours, dissolve by the heat of a water-bath, add the glycerine, and finally the colouring, and mix intimately.

The gelatine used must be of the finest quality, as the commoner kinds possess an unpleasant flavour. Other flavouring agents may be used in suitable proportions in place of orange-flower water, *e. g.* rose water, essence of almonds, oil or tincture of lemon or orange, raspberry juice, &c.

To prepare the pastils a suitable tin mould is required. Perhaps the best form is a diamond-shaped flat-bottomed tray, with vertical sides, each five to six inches long, and marked below into 100 equal parts by deeply indented lines running parallel to the sides. After thorough cleansing, the tray is oiled with the least possible quantity of almond oil, and carefully levelled. Four and a half ounces of the base is now melted on a water-bath, and the medicine thoroughly incorporated with it: if soluble it is simply added to the melted mass, and when dissolved poured out into the tray; if insoluble it is rubbed to a very fine powder, mixed with about an equal weight of glycerine in a mortar, and then mixed thoroughly with the melted base, pouring the whole into the tray just before it is cold enough to set. When quite cold the mass is removed, turned over on a slab, and cut with a knife in the lines marked by the divisions. The most important of the pastils in common use are those of the Throat Hospital Pharmacopœia, and those of cocaine and codeine.

		In each.	
Pastillus Acidi Borici,	containing	2 grains	boric acid.
,, ,, Carbolici	,,	½ grain	carbolic acid.
,, Ammonii Bromidi	,,	3 grains	bromide of ammonium.
,, ,, Chloridi	,,	2 ,,	chloride ,, ,,
,, Bismuthi	,,	3 ,,	subcarbonate of bismuth.

	In each.
Pastillus Bismuthi et Morphinæ containing	3 grains subcarbonate of bismuth. $\frac{1}{40}$ grain acetate of morphine.
„ „ et Potassii Chloratis, containing	3 grains subcarbonate of bismuth. 2 „ chlorate of potassium.
„ Iodoformi „	1 grain iodoform.
„ Cocainæ „	$\frac{1}{2}$ „ hydrochlorate of cocaine.
„ Codeinæ „	$\frac{1}{8}$ „ codeine.

Medicated Gelatines.—*Gelatines,* introduced by Dr Unna, of Hamburg, are used for outward application where very slow absorption is required; at the same time the film of gelatine forms a good protective coating which does not materially retard evaporation of the perspiration. Two bases are usually employed, the common zinc gelatine and the hard zinc gelatine, as follows:

	Common.	*Hard.*
Oxide of zinc	3 parts	2 parts.
Gelatine	3 „	6 „
Glycerine	5 „	6 „
Water	9 „	6 „

The gelatine is soaked and dissolved as usual in the water, the oxide of zinc rubbed thoroughly smooth with the glycerine, and the two mixed while warm, stirred till it begins to thicken, and allowed to cool in moulds or jars. They are applied by melting by a gentle heat, and painting on the skin with a camel's-hair pencil. Other medicaments may be added to the above bases; the hard base is used for combination with carbolic or salicylic acid, resorcin, naphthol, creasote, or potassium sulphide, which may be added up to 10 per cent.; the ordinary base is employed for most other substances, but tannin, pyrogallol, and oxide of mercury are incompatible.

Salve Mulls and Plaster Mulls.—*Salve mulls* and *plaster mulls* were also introduced by Dr. Unna; the former of these consist of a basework of mull or undressed muslin, impregnated on one or both sides with an ointment consisting of lard, lanoline, vaseline, or other fat, and kept in position by a bandage of mull; the latter consists of mull, covered on one side with gutta-percha tissue, the medicament being evenly spread on the latter. There is no plaster mass in the ordinary sense of the term, such substances as resin, turpentine, &c., being rigidly excluded, and replaced by pure india rubber or oleate of aluminium, which must be used in *only just sufficient quantity* to bring the active medicament to an adhesive consistence at the body temperature. They are prepared containing a definite quantity of active medicament to the square metre: for example, salicylic acid plaster mull may contain 40 grms. per square metre; that is to say, 40 grms. of the acid are spread over a square metre by the aid of a minimum quantity of medium.

Tabellæ.—Tabellæ (tablets) are prepared either by compression, when they are called compressed tablets, or in the form of a plastic mass which is afterwards divided by suitable means and dried; these are sometimes called *tablet triturates.*

Compressed tablets are made in a special machine, the finely powdered drug being fed into a cylinder in which it is compressed by dies to the required form.*

The size of the tablet is regulated by the use of different dies; and if a tablet is required containing a dose intermediate between the regular sizes, sugar of milk may be used as a diluent to produce a tablet of the next size larger. The common sizes vary from ½ to 5 grains, the form being lenticular, *i. e.* doubly convex discs. Unless very great power is employed it is necessary to use a small proportion of some excipient, so as to produce a slightly coherent powder, *but not a mass.* The excipients which will be found most useful are weak syrup, water, dilute alcohol, mucilage of starch, mucilage of tragacanth, solution of dextrine, glycerine of starch, weak gelatine solution, and white vaseline ; these are used with or without the addition of sugar of milk. The dies should be lightly dusted with talc to prevent adherence of the tablets. A matter of great importance is that the finished tablet should be readily dissolved or broken down under the influence of water. With soluble drugs like phenazone and chloride of ammonium this presents no difficulty ; but with such insoluble substances as sulphonal and phenacetine, unless a very soluble excipient be employed the tablets will remain unacted on by water for a considerable period. In such cases glycerine of starch, or gelatine solution with some powdered sugar, will be found suitable. For quinine and some other dry powders an ethereal solution of soft paraffin is advantageous. Sometimes, when the dose is very small, *e. g.* cocaine, arsenious acid, or the hypodermic tablets, it is necessary to use a large proportion of sugar of milk, powdered sugar, or sodium chloride to increase the bulk to the size of a half-grain or one-grain tablet. The hypodermic tablets are made to contain sufficient of the active substance for one injection, and must be rapidly soluble in water.

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* Some of these tablet machines are very elaborate and expensive constructions. Amongst the simplest and most ingenious, and withal the smallest and least costly that I have seen, is one which only weighs about 60 lbs., and stands on a base of 6 × 12 inches, and 12 inches high. This handy little machine is equal to the manufacture of tablets of various weights and diameters, which are of equal weight and hardness, beautiful appearance, and are turned out at the rate of from 40 to 60 per minute. The powder is placed in a hopper, from which it is "fed" into the cylinder, passed under the compressing punch, ejected and cast down a shoot, one tablet being produced at every second revolution of the fly-wheel. The weight or hardness of the tablet can be increased or diminished with ease, and the machine is adapted to take dies of various sizes, so as to produce tablets varying from $\frac{3}{16}$ to $\frac{5}{8}$ inch in diameter.

Tablet triturates, which were introduced in America by Dr Thomas Fuller, are really of the nature of lozenges, but much smaller, and commonly more concentrated ; they present the advantage of being more readily soluble than the compressed tablets. To prepare these tablets, the active ingredient in the form of powder, fluid extract, or other suitable preparation, is thoroughly well triturated with finely powdered sugar of milk, with or without the addition of a smaller proportion of sugar, gum acacia, or gum tragacanth ; then made into a mass with alcohol, water, dilute alcohol, or mucilage of starch. The mass should be just stiff enough to cohere by gentle pressure, but not too soft. This mass is then filled into a number of holes in a perforated metal, glass, or hard rubber plate, resting on a flat plate of the same material ; this is done by spreading it over the plate firmly with a silver or nickel plated spatula. The perforated plate is then slid off the other plate and turned over, to see if the holes are completely filled; if not, more of the mass is spread on this side in order to fill them. The tablets must now be removed and dried, which may be done by means of another plate bearing a number of pegs corresponding with the perforations in the first plate. These pegs serve to force out the moulded tablets and to support each in a position favorable to rapid drying, and the tablets are allowed to remain thus supported until they are hard enough for handling. Another plan is to force out the tablets into a tray or sieve, in which they are dried by exposure to a draught of dry air, and finally to a low temperature in the drying cupboard. A sieve is by far the best to dry them in, as it permits of evaporation of moisture from the under surface as well as the upper.

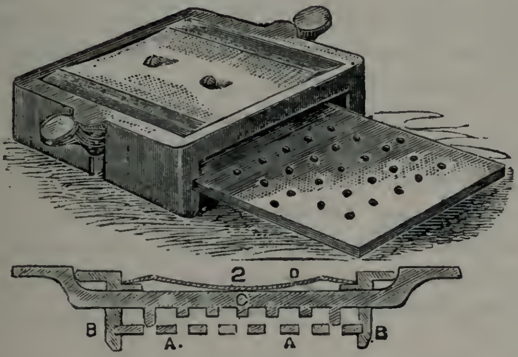

Fig. 76.—Tablet triturate Machine.

Andersen's apparatus, shown in Fig. 76, is a convenient form for general use, and the section (2) explains its construction, the projections in plate C being pressed into the perforations in plate A A to remove the tablets, B B being the framework. The tablets so made are small discs, but they could be made of other forms by altering the shape of the perforations, although the lenticular form which is popular for *compressed* tablets cannot be produced in this way. The tablets may be flavoured with oil of lemon, almonds, &c., and sweetened with saccharin or glycyrrhizin if too bulky for sugar.

Official Tabellæ.—Another form of base is cocoa, which is employed in the only official tabellæ, viz. those of nitroglycerine, which are described as "tablets of chocolate, each weighing two and a half grains, and containing one hundredth of a grain of pure nitro-glycerine" (trinitrin). The following formula may be used :

Take of—

Pure nitro-glycerine	1 grain.
Oil of theobroma	19 grains.
Cocoa (pure)	115 „
Powdered sugar	110 „
„ tragacanth	2½ „
Distilled water	10 minims.

Melt the oil of theobroma, and in this dissolve the nitroglycerine ; add the solution to the cocoa, previously melted by the heat of a water-bath. Mix the sugar and tragacanth in a mortar, and add the melted cocoa, &c. ; mix thoroughly, then add the water, and again mix. As soon as the mass becomes of a suitable consistence roll out in a mixture of sugar and starch, and divide into 100 tablets, either by the machine or as directed for lozenges.

Other tablets may be prepared in a similar manner with cocoa as a base, but in most cases it is not necessary to use oil of theobroma as a solvent ; it is used in this case because it prevents evaporation of the slightly volatile nitroglycerine.

Mr. H. Wyatt has suggested a simple method of dividing tablets, viz. to "pipe" the mass on a pill machine, and divide by simply pressing the cutter down on the piped mass; this gives fairly equal division, but the form is of course not the usual disc ('C. and D.,' i, 1888, p. 246).

Lamellæ.—Lamels, or ophthalmic discs, are very thin discs of gelatine, with some glycerine, each weighing about $\frac{1}{50}$ grain. They are employed in ophthalmic practice for dilating or contracting the pupil, or as a local anæsthetic, and also for the extemporaneous production of hypodermic injections. There are three official examples :

Lamellæ Atropinæ, containing $\frac{1}{5000}$ grain of sulphate of atropine.

Lamellæ Cocainæ, containing $\frac{1}{200}$ grain of hydrochlorate of cocaine.

Lamellæ Physostigminæ, containing $\frac{1}{1000}$ grain of physostigmine.

CHAPTER XXV

DEFINITIONS

The definitions of the various classes of galenical and other pharmaceutical preparations given in this chapter are intended to be used for reference as examples occur in the practical studies of the student, at which stage each one should be committed to memory, or at least its meaning thoroughly grasped. Many of the names included here do not occur in the preceding chapters, as they are of limited application, or do not refer to strictly pharmaceutical preparations; they are included, however, in order to make the glossary as complete as possible.

Abstract (*Abstractum, i,* N., 2nd).—A pulverised solid preparation, consisting of the soluble portion of a drug mixed with sufficient sugar of milk to bring the finished article up to one half of the original weight of the drug. Eleven are official in the U. S. P., the drugs being exhausted by alcohol.

Acetum, v. Vinegar.

Æther, v. Ether.

Ætheroleum = a Volatile Oil, *q. v.*

Alcohol (*Alcohol, olis,* N., 3rd).—A derivative of a hydrocarbon in which one or more atoms of H are replaced by an equal number of molecules of HO.

Alkaloid (*Alkaloideum, ei,* N., 2nd).—An organic substance possessing basic properties; usually derived from quinoline, pyridine, or uric acid, and obtained from a vegetable drug.

Apozema, atis, N., 3rd (Fr. *Apozème*).—A strong infusion or decoction prepared extemporaneously.

Aqua, v. Water (medicated).

Aqua pulverisata = Nebula, a spray, *q. v.*

Balneum, v. Bath.

Balsam (*Balsamum, i,* N., 2nd).—A natural product consisting of a resin or gum-resin, and containing benzoic or cinnamic acid.

Bath (*Balneum, ei,* N., 2nd).—Water, or water containing a small proportion of an active medicine, at a specified temperature.

Bougie (*Bouginarium, ii,* N., 2nd).—A slender, nearly cylindrical rod, intended for the local application of drugs to the nostril or urethra. Nasal bougies are commonly made 2 to 2½ inches in length; male urethral bougies, 2½ to 6 inches; and female urethral bougies, 1½ inches long.

Cachet, or Wafer.—A hollow biconvex body consisting of wafer-paper, and enclosing an active medicine.

Capsule (*Capsula, æ,* F., 1st).—A thin membrane, commonly of gelatine, enclosing an active drug.

Carbasus, v. Gauze.

Cataplasma, v. Poultice.

Caustic (*Causticum, i,* N., 2nd).—A solid or liquid preparation intended to cause local destruction of tissue.

Cerate (*Ceratum, i,* N., 2nd).—An unctuous preparation, softer than a plaster, but firmer than an ointment.

Charta, v. Paper.

Clyster = Enema, *q. v.*

Collodion (*Collodium, ii,* N., 2nd).—A solution of pyroxylin in an ethereal liquid, and usually containing some active principle. It is applied locally, and upon evaporation leaves a film which protects the part from the air and dust, at the same time allowing slower absorption than is the case with an ointment or liniment.

Collunarium, v. Lotion.

Collyrium, v. Lotion.

Confection (*Confectio, onis,* F., 3rd), or *Electuary* (*Electuarium, ii,* N., 2nd).—A soft paste containing one or more active ingredients, brought to the proper consistence by admixture with syrup, honey, or sugar. A *conserve* is a similar preparation, containing enough sugar to keep it from fermentation or other decomposition.

Cordial (*Cordiale, is,* N., 3rd).—A thin syrup, commonly flavoured with an essential oil or a fruit juice.

Decoction (*Decoctum, i,* N., 2nd).—A liquid preparation made by boiling the drug with water, and straining.

Draught (*Haustus, ûs,* M., 4th), *v.* Mixture.

Electuary (*Electuarium, ii,* N., 2nd) = Confection, *q. v.*

Elæo-saccharum, v. Oleo-saccharum.

Elixir (*Elixir,* N., indecl.).—A weak alcoholic preparation, usually sweetened with syrup, saccharin, or glycerine, and flavoured with essential oils or fruit juices.

Embrocation (*Embrocatio, onis,* F., 3rd), *v.* Liniment.

Emplastrum, v. Plaster.

Emulsion (*Emulsio, onis,* F., 3rd).—A mixture consisting of a watery liquid and an oil or resin, rendered miscible on simple agitation by the presence of a third substance (usually a *gum*).

Enema (*Enema, atis,* N., 3rd).—A liquid preparation designed for administration *per rectum.* Mucilage of starch is the usual vehicle. If the object is to obtain absorption or continued local action, it should be warm and the quantity small, *e. g.* Enema Opii or nutrient enemata; if, however, rapid evacuation of the bowels is desired the enema should be cold and in considerable quantity, *e. g.* Enema Magnesii Sulphatis and Enema Aloës.

Essence (*Essentia, æ,* F., 1st).—A strong solution of a volatile oil in spirit. The official examples are both 1 of oil in 4 of S. V. R.

Ether (*Æther, eris,* N., 3rd).—An organic compound bearing the same relation to an alcohol that a metallic oxide does to its hydrate. The term is sometimes applied, however, to express any very volatile liquid.

Extract (*Extractum, i,* N., 2nd).—A strong preparation, made by reducing the soluble portion of a drug (in water, spirit, &c.) to a solid or semi-solid consistence. A *liquid* or *fluid extract* is similarly prepared, but the concentration is only carried to the point of producing a powerful liquid preparation, usually of such a strength that 1 fl. part represents one part of the drug.

Fomentation (*Fomentum, i,* N., 2nd; and *Fotus, ûs,* M., 4th), *v.* Lotion.

Fumigation (*Fumigatio, onis,* F., 3rd; *Suffumigatio, onis,* F., 3rd).—A preparation which is used by igniting, and then inhaling the vapours which arise.

Fumus, i, = Fumigation, *q. v.*

Gargle (*Gargarisma, atis.*, N., 3rd), *v.* Lotion.

Gauze (*Carbasus, i*, F. 2nd).—A thin, cotton, net-like fabric, impregnated with an antiseptic medicament.

Gelatine (*Gelatina, æ*, F., 1st,).—A base consisting of gelatine, glycerine, and water, medicated by addition of antiseptic and other remedies. Melted before use, and applied in a thin film by means of a camel's-hair pencil.

Glycerine (*Glycerinum, i*, N., 2nd) , also called *Glycerole*, *Glycerite* (*Glyceritum, i*, N., 2nd).—A solution of an active medicine (usually an astringent, antiseptic, or emollient) in glycerine, sometimes slightly diluted with water. The glycerine is of value from its adhesive properties, thus securing prolonged contact with the affected part, without permitting any great evaporation of volatile drugs. Glycerines are commonly employed by local application with a brush, although the name is also sometimes applied to preparations intended for internal administration like syrups.

Gossypium, ii, v. Wool.

Granule, v. Pill.

Haustus, v. Mixture.

Honey (*Mel, mellis*, N., 3rd).—Medicated honeys are thick liquids containing a large proportion of honey. *Oxymels* (*Oxymellita*) contain acetic acid in addition.

Hydrolatum, v. Water.

Infusion (*Infusum, i*, N., 2nd).—An aqueous preparation, made, without boiling, by subjecting a crude drug to the action of water for a specified time, and straining. Boiling water is usually poured over the drug, and occasionally stirred whilst cooling.

Inhalation (*Vapor, oris*, M., 3rd).—A solution or admixture of volatile drugs, which is added to boiling or hot water contained in a suitable apparatus, so that the steam which arises may be inhaled, in order to obtain local action on the air-passages. The liquid used is also called in dispensing, inhalation drops (*Instillatio, onis*, F., 3rd).

Injection (*Injectio, onis*, F., 3rd).—A lotion (*q. v.*) applied to internal organs or membranes by means of a syringe.

Injection, hypodermic (*Injectio hypodermica*).—A powerful solution of an alkaloid, &c., which is used by injecting it under the skin by means of a specially constructed syringe, so as to introduce the medicine more rapidly into the blood. The syringe is usually of twenty minims capacity, graduated, and furnished with a needle-like orifice.

Instillatio, onis (F., 3rd).—Inhalation drops; *v.* Inhalation.

Insufflation (*Insufflatio, onis,* F., 3rd).—A fine powder applied to the throat, nose, ear, &c., by being blown in.

Juice (*Succus, i,* M., 2nd).—The liquid expressed from a fresh plant or part of a plant, to which one third its volume of rectified spirit has been added as a preservative.

Lamel (*Lamella, æ,* F., 1st).—A very thin disc of gelatine with some glycerine, containing an active substance; used by application to the eye in ophthalmic practice. Sometimes also used to produce hypodermic injections by solution in water.

Linctus (*Linctus, ûs,* M., 4th).—A medicine brought to the consistence of soft honey by the addition of sugar, mucilage, honey, &c., so that it can be taken by "licking" off a spoon; sometimes called a *lohoch.*

Liniment (*Linimentum, i,* N., 2nd).—A liquid or semi-liquid preparation used to rub or paint on a part for the purpose of producing local action, usually stimulation, or to allay pain. Those which are rubbed in are also sometimes called *embrocations;* and those which are applied by a brush, *pigments* or *paints.*

Lint (*Linteum, ei,* N., 2nd).—A medicated lint is lint impregnated with an antiseptic.

Liquid extract, v. Extract.

Liquor, v. Solution.

Lohoch = Linctus, *q.v.*

Lotion (*Lotio, onis,* F., 3rd).—A solution or mixture of active substances in water, for external application. A lotion made with boiling or hot water, and used hot, is called a *fomentation* (*Fomentum, i,* or *Fotus, ûs*). Lotions applied to particular parts of the body have received special names, *e. g.* (*Collyrium, ii,* N., 2nd), an eye wash; (*Collunarium, ii,* N., 2nd), a wash for the nostrils; (*Gargarisma, atis,* N., 3rd), a *gargle* or wash for the throat; and (*Injectio, onis,* F., 3rd) *injection,* a lotion applied to internal organs by a syringe.

Lozenge (*Trochiscus, i,* M., 2nd).—A hard disc or other conveniently shaped mass, consisting of a saccharine or similar basis mixed with active medicament, intended to be slowly sucked, so as in many cases to obtain local action on the throat and neighbouring parts.

Mass (*Massa, æ,* F., 1st).—A stiff semi-solid, consisting of an active substance or substances and excipient, from which pills are rolled.

Mel, v. Honey.

Mixture (*Mistura, æ,* F., 1st).—Mixtures are liquid preparations of varying character, containing much water, and designed for internal administration by the mouth. When dispensed in single doses a mixture is usually called a *draught* (*Haustus, ûs,* M., 4th; or *Potus, ûs,* M., 4th).

Mucilage (*Mucilago, inis,* N., 3rd).—A thick solution of a gum or similar substance, used for suspending heavy powders, oils, &c., or to thicken linctuses.

Mull, v. Plaster Mulls and Salve Mulls.

Nebula, v. Spray.

Oil (*Oleum, i,* N., 2nd).—Oils are of two kinds, *fixed* or *fatty,* and *volatile* or *essential.*

Fixed oils are greasy liquids which are decomposed by the action of heat, and cannot be distilled unchanged by any known means; chemically they mostly consist of the glycerine compounds of oleic, stearic, lauric, and some other organic acids.

Volatile oils, on the other hand, are readily distilled, either alone or by steam, with little or no change in their composition. They usually possess a powerful odour, and are nearly insoluble in water. They vary greatly in chemical composition; some are mainly hydrocarbons, others aldehydes, others oxidised hydrocarbons, &c. They are nearly all liquids, also called *Ætherolea.*

Ointment (*Unguentum, i,* N., 2nd).—A mixture or solution of one or more active substances in a soft basis, such as lard, which melts at or near the temperature of the body, and used as an external application. Used either by simply smearing or painting on the part, or by spreading on lint and applying as a bandage. Also called a salve.

Oleate (*Oleatum, i,* N., 2nd).—A solution of a basic substance in oleic acid.

Oleo-resin (*Oleo-resina, æ,* F., 1st).—A mixture of oil and resin obtained by evaporation of an ethereal tincture of a drug.

Oleo-saccharum (*i*, N., 2nd), also called *Elæo-saccharum.*—A mixture of a volatile oil with sugar.

Oleum, *v.* Oil.

Oxymel, *v.* Honey.

Paper (*Charta*, *æ*, F., 1st).—The official papers consist of paper coated on one side with an active medicinal substance, and employed to produce vesication or rubefaction. The term is also applied to a class of preparations consisting of bibulous paper impregnated with active drugs, and used by burning, and inhaling the vapours produced. It is better, however, to call these last by the name *fumus*, a fuming inhalation; or *fumigatio*, a fumigation.

Pastil (*Pastillus*, *i*, M., 2nd).—A base of gelatine and glycerine, medicated. Used as a lozenge.

Pessary (*Pessus*, *i*, M., 2nd).—A cone, consisting of a basis of low melting-point and an active substance, designed for administration *per vaginam.*

Pigmentum, *v.* Liniment.

Pill (*Pilula*, *æ*, F., 1st).—A small round or oval mass which can be conveniently swallowed, consisting of an active medicine or medicines and an excipient. Small pills, 1 grain or less, are sometimes called by the diminutive term *pilules*; sometimes also *granules*, or *globules*.

Plaster (*Emplastrum*, *i*, N., 2nd).—An adhesive substance spread upon leather or other suitable material, designed to obtain local or slow constitutional action by adhesion to the body.

Plaster Mull.—Undressed muslin attached to gutta-percha tissue, and spread with an active medicament by the aid of a small proportion of a non-irritant medium.

Potus = a potion or draught, *v.* Mixture.

Poultice (*Cataplasma*, *atis*, N., 3rd).—A soft semi-solid, used as a means of applying heat and moisture, with or without active medicinal agents.

Powder (*Pulvis*, *eris*, M., 3rd).—The official powders are intimate admixtures of two or more finely pulverised drugs.

Ptisana (*æ*, F., 1st) (Fr. *tisane*) = Infusion, *q. v.*

Pulp (*Pulpa, æ,* F., 1st).—A soft semi-solid, prepared from a plant or part of a plant (usually the fruit), and containing all except the most woody or fibrous portions, which are separated by means of a sieve.

Pulvis, v. Powder.

Resin (*Resina, æ,* F., 1st).—A principle or mixture of principles obtained from certain drugs by extraction with rectified spirit and removal of saccharine and other substances by washing with water ; or by distilling the essential oil from natural oleo-resins.

Rotula (*æ,* F., 1st).—A small round lozenge of melted sugar, serving as a base for various essential oils.

Salve = Ointment, *q. v.*

Salve Mull.—Undressed muslin spread on one or both sides with an ointment.

Solution (*Liquor, oris,* M., 3rd).—A clear aqueous liquid containing a definite proportion of an active medicine.

Spirit (*Spiritus, ûs,* M., 4th).—An alcoholic solution of a volatile substance. Most of the spirits are simple solutions of volatile oils, but others are distilled.

Spray (*Nebula, æ,* F., 1st).—An aqueous solution applied in a very finely divided condition by means of a special apparatus. Also called *Aqua Pulverisata,* and spray inhalations.

Succus, v. Juice.

Suffumigatio (*onis,* F., 3rd), *v.* Fumigation.

Suppository (*Suppositorium, ii,* N., 2nd).—A small cone consisting of a basis of low melting-point and an active substance, designed for administration *per rectum,* to obtain local action, or to avoid introduction of medicines into the stomach.

Syrup (*Syrupus, i,* M., 2nd).—An aqueous solution or liquid extract of a drug, thickened and sweetened with a large proportion of sugar.

Tablet (*Tabella, æ,* F., 1st).—Tablets consist either of pure or nearly pure drugs compressed into small discs or biconvex masses ; or of active drugs mixed with a saccharine or chocolate basis, and formed into discs similar to, but much smaller than lozenges.

Tincture (*Tinctura, æ,* F., 1st).—A tincture is a spirituous solution of a drug, or of the soluble portion of a drug, much weaker than a liquid extract.

Triturate (*Trituratio, onis,* F., 3rd).—A powder consisting of an active ingredient mixed with a definite proportion of a diluent, usually sugar of milk. Compound elaterin powder of the Pharmacopœia is really a triturate.

Trochiscus, v. Lozenge.

Unguentum, v. Ointment.

Vapor, v. Inhalation.

Vaseline (*Vaselinum, i,* N., 2nd).—An ointment of which vaseline is the base.

Vinegar (*Acetum, i,* N., 2nd).—A solution of the soluble portions of a drug in acetic acid.

Vinum, v. Wine.

Water (*Aqua, æ,* F., 1st).—Medicated waters are dilute solutions of aromatic substances (usually essential oils) in distilled water; they are commonly made by distilling the water in contact with aromatic drugs (*Hydrolata*).

Wine (*Vinum, i,* N., 2nd).—A solution of the soluble parts of a drug in wine.

Wool (*Gossypium, ii,* N., 2nd).—Absorbent cotton impregnated with an antiseptic substance, used in surgery.

Many of these definitions are taken from a paper by Mr J. Ince, published in the 'Pharm. Journ.' [3], xxii, p. 669.]

APPENDIX

Table of Comparison of Fahrenheit's and Centigrade Thermometers

Fahrenheit.	Centigrade.	Fahrenheit.	Centigrade.	Fahrenheit.	Centigrade.
0	−17·7	64·4	18	125·6	52
+3·2	16	66·2	19	127·4	53
5·0	15	68	20	129·2	54
6·8	14			131	55
8·6	13	69·8	21	132·8	56
10·4	12	71·6	22	134·6	57
12·2	11	73·4	23	136·4	58
14	10	75·2	24	138·2	59
		77	25	140	60
15·8	9	78·8	26		
17·6	8	80·6	27	141·8	61
19·4	7	82·4	28	143·6	62
21·2	6	84·2	29	145·4	63
23	5	86	30	147·2	64
24·8	4			149	65
26·6	3	87·8	31	150·8	66
28·4	2	89·6	32	152·6	67
30·2	1	91·4	33	154·4	68
32	0	93·2	34	156·2	69
		95	35	158	70
33·8	+1	96·8	36		
35·6	2	98·6	37	159·8	71
37·4	3	100·4	38	161·6	72
39·2	4	102·2	39	163·4	73
41	5	104	40	165·2	74
42·8	6			167	75
44·6	7	105·8	41	168·8	76
46·4	8	107·6	42	170·6	77
48·2	9	109·4	43	172·4	78
50	10	111·2	44	174·2	79
		113	45	176	80
51·8	11	114·8	46		
53·6	12	116·6	47	177·8	81
55·4	13	118·4	48	179·6	82
57·2	14	120·2	49	181·4	83
59	15	122	50	183·2	84
60·8	16			185	85
62·6	17	123·8	51	186·8	86

Fahrenheit.	Centigrade.	Fahrenheit.	Centigrade.	Fahrenheit.	Centigrade.
188·6	87	248	120	419	215
190·4	88	257	125	428	220
192·2	89	266	130	437	225
194	90	275	135	446	230
		284	140	455	235
195·8	91	293	145	464	240
197·6	92	302	150	473	245
199·4	93	311	155	482	250
201·2	94	320	160	491	255
203	95	329	165	500	260
204·8	96	338	170	509	265
206·6	97	347	175	518	270
208·4	98	356	180	527	275
210·2	99	365	185	536	280
212	100	374	190	545	285
		383	195	554	290
221	105	392	200	563	295
230	110	401	205	572	300
239	115	410	210		

Table of Specific Gravity of Spirit of various strengths, with equivalents in Rectified Spirit, 56 o. p., and Proof Spirit to nearest whole number.

Sp. gr. at 15·5° C.	Alcohol % by weight.	Rectified spirit % by volume.	Proof spirit % by volume.	Sp. gr. at 15·5° C.	Alcohol % by weight.	Rectified spirit % by volume.	Proof spirit % by volume.
·7938	100·0	112·66	175·25	·895	60·3	77	119
·795	99·6	112	175	·900	58·0	74	115
·800	98·0	111	173	·905	55·9	72	112
·805	96·4	110	171	·910	53·6	69	108
·810	94·6	109	169	·915	51·4	67	104
·815	92·8	107	167	·920	49·2	64	100
·820	91·0	106	165	·925	46·9	62	96
·825	89·2	104	162	·930	44·6	59	92
·830	87·2	103	160	·935	42·3	56	87
·835	85·3	101	157	·940	39·8	53	83
·838	84·08	100	155·55	·945	37·1	50	77
·840	83·3	99	154	·950	34·5	47	72
·845	81·4	98	152	·955	31·6	43	67
·850	79·3	96	149	·960	28·6	39	61
·855	77·3	94	146	·965	25·1	34	54
·860	75·1	92	143	·970	21·3	29	46
·865	73·0	90	139	·975	17·2	24	37
·870	70·8	88	136	·980	13·1	18	29
·875	68·8	85	133	·985	9·4	13	20
·880	66·7	83	130	·990	5·9	8	13
·885	64·6	81	126	·995	2·8	4	6
·890	62·4	79	123	1·000	0·0	0	0

Spirit of any desired specific gravity above ·838 can be made by measuring the required amount of rectified or proof spirit, according to the above table, and adding sufficient distilled water *to produce* the desired quantity of the diluted spirit.

Twaddell's Hydrometer.

To convert the degrees of Twaddell's hydrometer into specific gravity compared with water as 1000, multiply by 5 and add 1000.

Equivalents of Grammes in Grains.

Grms.	Grains.	Grms.	Grains.	Grms.	Grains.
1	15·432	4	61·729	7	108·026
2	30·865	5	77·161	8	123·458
3	46·297	6	92·594	9	138·891

Equivalents of Grains in Grammes.

Grains.	Grms.	Grains.	Grms.	Grains.	Grms.
1	0·0648	4	0·2592	7	0·4536
2	0·1296	5	0·3240	8	0·5184
3	0·1944	6	0·3888	9	0·5832

From these tables the equivalent in grains of any number of grammes, or *vice versâ,* may readily be ascertained by simple addition, after removal of the decimal point to left or right as required, *e. g.*—

5·31 grammes = 77·161 + 4·6297 + ·1543 = 81·9450 grains.

Various Useful Data.

The following formulæ will frequently prove useful to the pharmacist:

To find the area of a circle:

Let A = area.
R = radius.
π = 3·1416.
Then A = πR^2.

To find the contents of a sphere :

Let C = contents.
Then $C = 4{\cdot}1888 \cdot R^3$.

To find the contents of a cylindrical vessel :

Let C = contents.
Then C = area of the base × height.

To find the contents of a rectangular vessel :

Let C = contents.
A = length of one side.
B = ,, other side.
H = height.
Then $C = A \times B \times H$.

To find the weight of any element contained in a given weight of a compound:

Let W = weight of compound.
M = molecular weight of compound.
A = atomic weight of element sought.
N = number of atoms of the element in a molecule of the compound.
x = weight sought.

$$\text{Then } x = \frac{A \times N \times W}{M}.$$

To find the weight of any substance required to yield a given weight of some other substance :

1st. Write equation expressing reaction.
2nd. Let W = weight of substance given.
M = molecular weight of substance produced × number of molecules indicated by equation.
N = molecular weight of substance required × number of molecules indicated by equation.
x = weight sought.

$$\text{Then } x = \frac{W \times N}{M}.$$

Freezing Mixtures.

The following freezing mixtures may be used for operations of refrigeration :

Snow	3 parts.
Crystallised calcium chloride	4 ,,

Snow	8 parts.
Hydrochloric acid	5 ,,

Snow, or broken ice	12 parts.
Salt	5 ,,
Ammonium nitrate	5 ,,

Snow, or broken ice	5 parts.
Salt	2 ,,
Ammonium chloride	1 part.

Sodium sulphate	8 parts.
Hydrochloric acid	5 ,,

Snow, or broken ice	2 parts.
Salt	1 part.

Ammonium nitrate	1 part.
Water	1 ,,

Ammonium chloride	1 part.
Potassium nitrate	1 ,,
Water	3 parts.

Freezing mixtures should be kept well stirred to obtain the full effect.

ADDENDA

Page 68. Add to table—

Acidum Carbolicum liquefactum: Carbolic acid, 10; water, 1. 1·065. By gentle heat.

Page 69. Add to table—

Oxymel: Clarified honey, 8 parts; acetic acid and distilled water, of each 1 fl. part. By gentle heat.

Page 80. Add to table—

Cataplasma Carbonis: Wood charcoal, in powder, 1; bread crumb, 4; linseed meal, 3; boiling water, 20. Soak the bread in water ten minutes, add linseed and half the charcoal, and sprinkle other half on surface of poultice.

Page 118. Add to table—

Cataplasma Conii: Hemlock juice, 1 fl. part; linseed meal, 4 parts; boiling water, 10 fl. parts. Evaporate juice to one half, and stir this into linseed and water previously mixed.

Page 162. Add after granulated sulphate of iron—

Ferrum Redactum.

Precipitate ferric hydrate from solution of the perchloride as usual, wash, and dry. Introduce dry oxyhydrate into an iron tube, and heat to a full red heat whilst passing a current of dry hydrogen through the tube, obtained by action of dilute H_2SO_4 upon zinc, and dried by passing through strong H_2SO_4 and over chloride of calcium. When gas leaves tube as fast as it enters, the tube is cooled, the current of hydrogen stopped, and the reduced iron enclosed in well-stoppered bottles. $Fe_2O_22HO + 3H_2 = Fe_2 + 4OH_2$.

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